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Shale Swelling Inhibition Properties of (Carboxymethyl)trimethylammonium chloride (CTAC) in Water-based Drilling Fluids

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Abstract

One of the most important challenges during drilling an oil or gas well is dealing with shale formations and the subsequent shale swelling. In this study, we utilized Carboxymethyl Trimethyl Ammonium Chloride (CTAC) to inhibit shale swelling, representing a novel application for this particular cationic surfactant. Several experiments were carried out to assess the effectiveness of CTAC in preventing shale swelling and to gain insights into the underlying mechanisms. Moreover, based on the results, CTAC is highly effective at low concentrations and can be used with other common additives. Furthermore, the contact angle in the presence of CTAC, cetyltrimethylammonium bromide (CTAB), and potassium chloride (KCl) at 1 wt.% in the bentonite mixture was 77°, 75°, and 38°, respectively. Also, by adding CTAB, total shale recoveries in simple and complete drilling muds increased by 5.53% and 0.94%, respectively. Meanwhile, In the presence of CTAC, the increase was equal to 12.37% and 6.43%, respectively. In addition, CTAC and CTAB in the complete drilling mud reduced the swelling by 9.94% and 4.2%, respectively. Ultimately, comparative studies show that CTAC performs better as a new inhibitor than CTAB and KCl as conventional inhibitors.

Keywords: Drilling fluid; Surfactant; Wettability; Contact Angle; Shale Swelling; Bentonite.

Introduction

The process of extracting oil, gas, and gas hydrate reservoirs involves deep drilling, and water-based mud is a frequently employed type of drilling fluid because it can be chemically modified to suit specific needs [1, 2]. Drilling is the most challenging operation in the oil and gas extraction stages, especially when it reaches sensitive and reactive formations with drilling mud. Depending on the type of formation, changes in mud and drilling tools are necessary. In addition, there are huge unconventional hydrocarbon reservoirs in shale formations [3-6]. Moreover, shale drilling is one of the common challenges. Furthermore, the shale swells by absorbing water from the drilling mud, and with its collapse, the drilling operation faces a serious risk and other problems like formation damage [7-10]. Also, this incident causes many problems while driving the well-

logging tools and finally interpreting the well-logging graphs. Moreover, in some cases, it causes the well to be lost during drilling, which leads to side-tracking or well abandonment [11-13].

The importance of drilling shale formations increases when we know they contain significant oil in some areas [14]. In addition, oil-based muds can control swelling and prevent the collapse of shale formations. Still, they are not widely used due to their high cost and negative environmental impact, particularly in offshore drilling [15]. On the other hand, environmental protection organizations have imposed many restrictions on this type of drilling mud. In these ways, engineers have started using modified water-based drilling fluids to control swelling and prevent well instability during drilling.

Zhong et al. [16], by investigating the anti-swelling

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properties of polyether diamine, showed that the molecules of this amine can enter the interlayer structure of clay minerals and exchange ions with the cations between these plates when this polymer is added to water; it releases ammonium ions.

These ions can then be adsorbed onto the surface of clay minerals, which neutralizes their negative charge and significantly reduces the repulsive force between them. In other words, by establishing hydrogen bonds between the surface of clay plates and the molecules of this polymer, water molecules are removed from between the clay plates, and the distance between the clay plates is reduced, causing the dehydration of clays. A hydrophobic protective layer is formed when polyether diamine molecules are adsorbed onto the clay surface. This layer prevents water molecules from reaching the clay mineral plates.

The utilization CTAB as a shale stabilizer in drilling fluids was suggested by Moslemizadeh et al. [17]. In addition, when CTAB was present in an aqueous solution containing bentonite particles, the electric charge on the surface of the plates decreased. This caused the bentonite particles to accumulate and grow in size. CTAB adsorption onto the bentonite plate surface decreased interlayer water molecules, which played a significant role in reducing bentonite particle swelling. The mechanism of action of CTAB involved entering the interlayer structure of bentonite particle plates, displacement of primary cations between the plates, and alteration of plate surface wettability. Wettability alteration is generally one of the characteristics of surfactants. Due to their amphiphilic structure, they can alter the wettability of surfaces after being adsorbed on them [18-20]. Barati et al. [21] researched a new amine-based additive called Polyethoxylated tallow, which was added to drilling fluids to enhance shale stability and reduce swelling. The inhibitory properties of this material were investigated through several tests under different conditions, and it was found out that it prevents the swelling of bentonite particles and disintegration of drilling cuttings in aqueous environments. Furthermore, polyethoxylated tallow amine was evaluated against potassium chloride, a commonly used shale stabilizer in the drilling industry, and found to have similar performance. Furthermore, this additive was compatible with other additives commonly employed in drilling fluids. Moreover, the amine groups within the additive interact with the surface of bentonite particles through hydrogen bonding. As a result, water absorption is reduced, and ultimately, the swelling of bentonite particles is diminished. In addition, the effectiveness of Horsetail plant extract in controlling clay mineral swelling was examined. Moreover, the study found that this extract is compatible with other additives commonly used in drilling fluid and performs well, even when compared to sodium silicate and polyamine. In addition, the mechanism behind its performance involves establishing hydrogen bonds with the surface of clay mineral plates and decreasing the number of available sites on the surface of particle plates [22].

Also, some nanoparticles have been investigated as inhibitors of shale swelling in drilling mud. Akhtarmanesh et al. [23] investigated the impact of two types of nanoparticles, sized at 35 and 50 nm, on the efficiency of shale membranes. Moreover, the study revealed that these particles enter

the porous spaces of shale formations and obstruct the bottlenecks, reducing the formation's permeability. As a result, the infiltration of drilling fluid filtrate into the formation is decreased, while reverse osmosis flow from the shale is enhanced. Furthermore, these two effects contribute to the stability of shale formations. Moreover, CTAB-modified graphene was used in the modified water-based drilling mud. It achieved the highest shale dispersion recovery at 88.5%, and the swelling test demonstrated that clay materials maintain stability against the reactive effects of water when the inhibitor is present [24]. Cetyl Trimethyl Ammonium Chloride also showed excellent efficiency in reducing the filtration loss of drilling fluid. Moreover, when added at a concentration of 1.0%, it reduced filtration loss exceeding 60% [25]. In addition, this surfactant also had a great synergy with graphite to improve the lubrication performance of drilling fluid [26]. Hexadecyltrimethylammonium chloride from the same family was used as an additive in water-based drilling fluid to improve dispersion stability [27].

This research examined the effectiveness of (Carboxymethyl) trimethylammonium Chloride (CTAC) as a shale stabilizer. Moreover, the inhibitory performance of CTAC was evaluated under various conditions through different tests, including bentonite sedimentation and swelling inhibition, dynamic linear swelling, shale sample recovery, wettability, and zeta potential evaluations, as well as isothermal water absorption and thermogravimetric analysis (TGA). Furthermore, the compatibility of CTAC with other common drilling mud additives was investigated. Additionally, the shale stabilization performance of CTAC was compared to other stabilizers, such as potassium chloride (KCl) and CTAB, in the drilling industry.

Research on the types of shale swelling inhibitors is being developed; however, limited inhibitor cases have been investigated in these studies, and new inhibitors can be introduced to the existing literature to fill the data gap. Therefore, the novelty of this study is to investigate a new application of CTAC as a shale swelling inhibitor.

Materials and Methods

Shale and Bentonite Samples

Pieces of a shale core as a mineral containing clay minerals were extracted from a depth of 3559.60 meters in a well of one of the oil fields in Iran. Moreover, according to XRD analysis, the shale samples contained 3% dolomite, 2% calcite, 30% quartz, 19% muscovite-illite, 7% albite, 8% orthoclase, 24% kaolinite, 3% pyrite, and 1% anatase. Bentonite in powder form was obtained from Pars Drilling Company, Iran.

Drilling Mud Materials

The thickeners used in this study were starch and xanthan gum, while partially hydrolyzed polyacrylamide, low and regular viscosity polyanionic cellulose, alcoholic antifoam, and barite were acquired from Pars Drilling Company in Iran. Merck in Germany provided the sodium hydroxide (NaOH) and sodium chloride (NaCl) used in the experiments.

Shale Swelling Inhibitors

Merck, Germany provided common inhibitors such as

potassium chloride (KCl) and CTAB with the chemical formula of $C_{16}H_{33}N(CH_3)_3Br$, molecular weight of 365.45 g/mol, and a new inhibitor called CTAC. CTAC, with the formula of $C_5H_{12}ClNO_2$ and molecular weight of 167.64 g/mol, is a cationic surfactant that consists of a central nitrogen atom attached to three methyl groups ($-CH_3$) and a carbon atom attached to the carboxyl functional group ($-COOH$). CTAC, or betaine hydrochloride, is a versatile compound with various pharmaceutical and biomedical applications due to its properties as a cationic surfactant [28]. CTAC was tested alongside KCl and CTAB as a new shale stabilizer in this study. In Fig. 1, the molecular structures of CTAC and CTAB are shown.

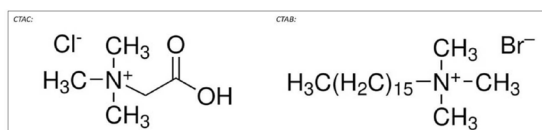


Fig. 1 The molecular structure of CTAC and CTAB.

The Performance Evaluation Experiments

Bentonite Sedimentation Test

When a clay mineral sample is added to an aqueous environment, its constituent plates are separated from each other and form a colloid. The resulting colloid will be unstable if the fluid around these plates has inhibitory properties. This process leads to the connection of clay plates and deposits. The Bentonite sedimentation test measures the degree of instability of clay minerals in inhibiting environments [29]. In this test, first, an aqueous solution containing a shale stabilizer was prepared. The stabilizers were used at a concentration of 5 wt% in this test. Then, a small amount of a clay mineral (bentonite) was added and mixed well. The resulting colloidal mixture was poured into test tubes and left to stand still. After a sufficient period (depending on the clay mineral's nature and the stabilizer's performance), a specific contact surface was created between the deposited bentonite and its upper solution. In addition, the lower this interface shows, the better the performance of the stabilizer.

The distance between the contact surface and the edge of the pipe cap (h) is measured, and then it is divided by the internal length of the pipe (H). Moreover, the result of this calculation, the h/H ratio, is recorded over time and analyzed for various concentrations of a shale stabilizer. Furthermore, the h/H ratio is expressed as a unitless value. Moreover, this test is performed in two ways: hydrated and dehydrated. Moreover, in the hydrated state, clay mineral (bentonite) is

first introduced into the aqueous medium, and after mixing and forming a colloidal mixture, the test shale inhibitor is added. In this case, the stability of the clay mineral is higher than in the dehydrated state because the bentonite particles have enough time to swell to form a colloidal mixture.

Bentonite Swelling Inhibition Test

When drilling formations with active clay minerals, the water-based drilling mud's rheological properties typically increase because there are active clays, and hydration occurs. To simulate this process, the bentonite swelling inhibition exam is performed [30].

In this test, solutions containing the shale stabilizer at different concentrations were prepared first, and then a certain amount of bentonite was added. Then it was placed in a stationary state under environmental conditions.

The rheological properties and filtrate volume are measured after 24 hours. Drilling fluid with lower rheological parameters, including apparent viscosity (AV), plastic viscosity (PV) and yield point (YP), and higher filtrate volume, indicates better inhibitory properties [17, 31]. The DV2T Touch Screen Viscometer (Brookfield, USA) was used to measure rheology. These properties can be calculated by Equations 1- 3 [32]:

$$AV = \frac{\theta_{600}}{2} \quad (1)$$

$$PV = \theta_{600} - \theta_{300} \quad (2)$$

$$YP = (2\theta_{300} - \theta_{600}) / 2YP = 2\theta \quad (3)$$

where PV and AV are in cP; YP in Pa, and θ_{600} and θ_{300} viscometer dial readings at angular velocities of 600 and 300 rpm, respectively.

The filtrate volumes of mixtures were determined using filter paper and a filtrate measuring device under controlled conditions of 100 pounds per square inch (psi) pressure (~ 0.689 MPa) and 25 degrees Celsius temperature over 30 minutes.

Compatibility Test

This test involves measuring the rheological changes and filtrate extracted from different drilling muds before and after the addition of the shale stabilizer [33]. To conduct this test, drilling muds were made based on Table 1 and heated at 70 °C in an oven (the UF55 model of Memmert company, Germany) for 4 hours before cooling to 55 °C. The above equations measured the rheological parameters, and the removed filtrate was measured under the conditions above [34, 35].

Table 1 The composition of drilling fluid.

Mud types	Composition	Density (kg/m ³)
Simple mud	Distilled water (93.9 wt%), PAC-LV (1.0 wt%), NaOH (2.5 wt%) and HV starch (2.6 wt%)	1008.534
Simple mud + shale stabilizer at 1 wt%	Distilled water (93.9 wt%), PAC-LV (1 wt%), NaOH (2.5 wt%) and HV starch (2.5 wt%)	1008.534
Complete mud	Distilled water (76.2 wt%), PAC-LV (0.4 wt%), PAC-R (0.3 wt%), PHPA (0.2 wt%), anti-alcoholic foam (0.04 wt%), Xanthan gum (0.2 wt%), NaCl (8.3 wt%), KCl (3.5 wt%), NaOH (2.1 wt%) and barite (8.7 wt%)	1184.332
Complete mud + shale stabilizer at 1 wt%	Distilled water (75.1 wt%), PAC-LV (0.4 wt%), PAC-R (0.3 wt%), PHPA (0.2 wt%), anti-alcoholic foam (0.04 wt%), Xanthan gum (0.2 wt%), NaCl (8.1 wt%), KCl (3.4 wt%), NaOH (2.0 wt%) and barite (9.1 wt%)	1184.332

Shale Sample Recovery Test

In this test, a specific amount of shale samples with a specific size and the studied fluid sample are placed in contact with each other at a specific temperature. After a certain period, the shale sample recovery is calculated using the gravimetric method [36].

First, shale samples were crushed, and 2-4 mm samples were collected by sieves. 350 ml of CTAC, CTAB, and KCl aqueous solutions or the drilling muds of Table 1, along with 10 grams of shale samples, were placed in the rotary oven cells at 70 °C for 16 hours. Then, it was cooled to 40 °C in the environment. Then, the cell contents were filtered with sieve No. 10, placed on sieve No. 35, and washed with 10 wt% KCl solution. The filtered shale samples by the two sieves were dried at 105 °C. Finally, Equation 4 was used for the shale samples recovery calculation for each fluid:

$$SR(\%) = \frac{W_{secondary}}{W_{Initial}} * 100\% \quad (4)$$

Dynamic Linear Swelling Test

This test is employed to assess the extent of shale swelling caused by the quantitative interaction with water-based drilling muds. Moreover, it helps to observe the response of clay mineral samples to various drilling muds. In this test, observing and evaluating the behavior of clay mineral samples in very short intervals (0 to 5 minutes) and long intervals (more than 360 minutes) is possible. In this test, shale samples extracted from cores or even drilling cuttings can be used. To prepare for the test, powdered clay mineral samples were compressed under 6000 psi (~41.368 MPa) of pressure for 30 minutes to form tablets. Then, the bentonite tablet was fixed inside the dynamic linear swelling device to measure its swelling in contact with the target fluid. In this test, it is possible to apply temperature and fluid circulation together. Furthermore, the output of this device includes graphs related to the clay sample swelling rate with time, as well as the graph of temperature changes during the test. The device's setup includes a magnetic stirrer, a thickness gauge and a bentonite tablet holding cell. Moreover, as the volume of shale samples decreases over time, the inhibitory power of drilling mud increases. Aqueous solutions of CTAC, CTAB, and KCl or the drilling mud mentioned in Table 1 were transferred into the cell to perform this test. In addition, the thickness of the bentonite tablet placed in the device started to increase in contact with the fluid. Equation 5 was used to calculate the swelling based on the changes in tablet thickness at any time:

$$Swelling(\%) = \frac{T_{inc}}{T_i} * 100\% \quad (5)$$

where T_i is initial thickness and T_{inc} is its increase in millimeter

Mechanistic Experiments

Wettability Test

Wu's method was utilized to qualitatively examine how a shale swelling inhibitor affects the alteration of the wettability of clay minerals [37]. In this method, solutions of stabilizers CTAC, CTAB, and KCl at 1 wt.% and bentonites at 0.5 wt.% were prepared, and then the surfaces of glass slides were smeared with them. After the slides had dried in the environment, drops of water were slowly placed on the slides' surfaces, and the wettability behaviors were observed visually.

If a stabilizer has not altered the wettability of bentonite, the water drop will be quickly absorbed by the layer on the surface of the glass slide. If the bentonite surface alters wettability, then the water droplet on the slide will either remain steady or be absorbed more slowly by the layer on the surface of the glass slide.

Zeta Potential Test

To determine the zeta potential, the stabilizer-modified bentonite solution was initially prepared. To begin with, a 1 wt.% bentonite solution was created and allowed to settle in a beaker under normal conditions for two days. The supernatant solution was centrifuged for one minute at 3000 rpm to remove any impurities. Next, the solutions were heated and dried at 50 °C. The outcome was modified bentonite in a powdered form, which was carefully weighed. This modified bentonite was then used to produce a 0.5 wt.% aqueous solution.

To proceed with the zeta potential measurement, a specific concentration of CTAC was mixed with 50 ml of the solution. Moreover, the mixture was subjected to magnetic stirring for 30 minutes and then shaken on a shaker for 24 hours to ensure proper mixing. Finally, the zeta potential of the resulting solution was determined using a zeta sizer device (ZS Nano Z model of Malvern Panalytical, UK). In addition, this measurement helps assess the modified bentonite solution's stability and surface charge, providing valuable insights into its potential applications.

Supernatant solution is typically not used for direct zeta potential testing because it may not accurately represent the zeta potential of the colloidal or dispersed particles of interest. Moreover, zeta potential measures the electrical charge and potential difference between particles and the surrounding medium in a colloidal system. Furthermore, it plays a crucial role in determining the stability and behavior of colloidal dispersions. In addition, to obtain accurate and meaningful zeta potential measurements, analyzing the actual dispersed particles without any alterations or disruptions to their state is important. Moreover, this can be achieved by testing the coherent colloidal suspension, avoiding any steps that may cause the sample's aggregation, sedimentation, or dilution. Ultimately, modern zeta potential analyzers are designed to measure particles' zeta potential while dispersed in their medium, providing reliable and representative results.

Isothermal Water Absorption Test

At first, CTAC-modified bentonite was prepared as in the previous step. Distilled water was added to a 250 ml beaker to conduct the isothermal water absorption experiment. Furthermore, a gram of altered bentonite was applied onto aluminum foil and then positioned over the water inside the beaker covered with cellophane at room temperature. In addition, the bentonite weight was measured at different time intervals using a scale, and the weight increase resulting from the water absorption was determined using the modified bentonite weight at the beginning.

Thermal Analysis

The heart of a TGA device is a precise balance with a programmable heating oven. The thermal analyzer model

STA PT1600 manufactured by LINSEIS Company was used to conduct TGA analysis on the modified bentonite. The analysis was conducted under a nitrogen gas flow, starting from 25°C to ending at 300°C, with a °C/min rate.

Results and Discussion

The Results of Performance Evaluation Experiments

The Results of the Bentonite Sedimentation Test

The outcomes of the sedimentation examination have been illustrated and shown in Figs. 2 to 7. Adding 0.5 wt.% of CTAC resulted in the inability of bentonite particles to create a dependable colloidal blend, causing precipitation. This indicates that at the lower concentration, CTAC inhibits bentonite particles effectively. Moreover, as the amine concentration was raised to 1 wt.%, the instability of the bentonite particles grew and caused accelerated sedimentation. In addition, a small rise in the bentonite particle instability was observed with concentrations of 3 wt.% or more, as shown in Fig. 3. Furthermore, the final state of sedimentation behavior of bentonite particles for CTAC amine in different concentrations is shown after 100 hours.

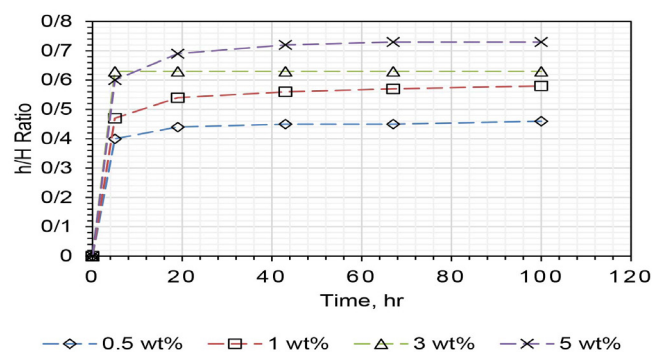


Fig. 2 Results of the bentonite sedimentation test in the presence of CTAC.

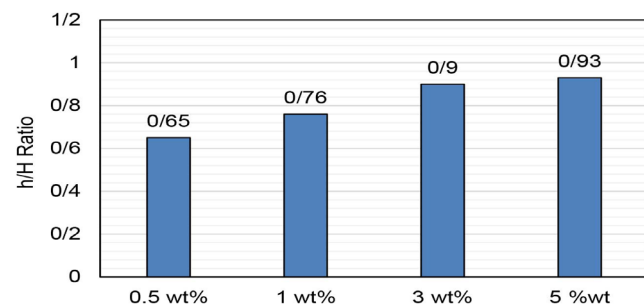


Fig. 3 Final results of the bentonite sedimentation test in the presence of CTAC.

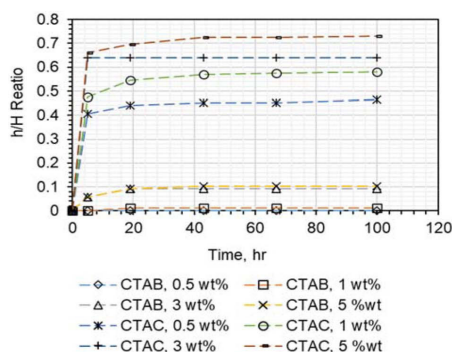


Fig. 4 Results of the bentonite sedimentation test in the presence of CTAC and CTAB.

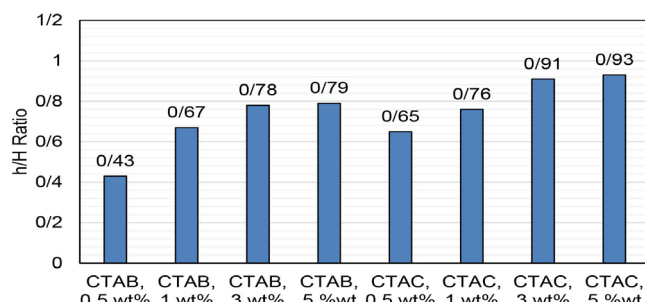
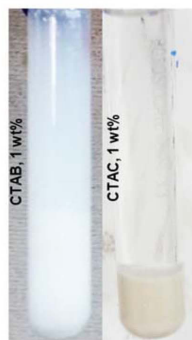


Fig. 5 Final results of the bentonite sedimentation test in the presence of CTAC and CTAB.

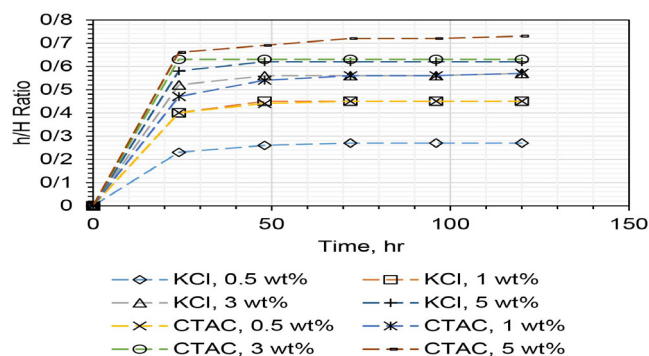


Fig. 6 Results of the bentonite sedimentation test in the presence of CTAC and KCl.

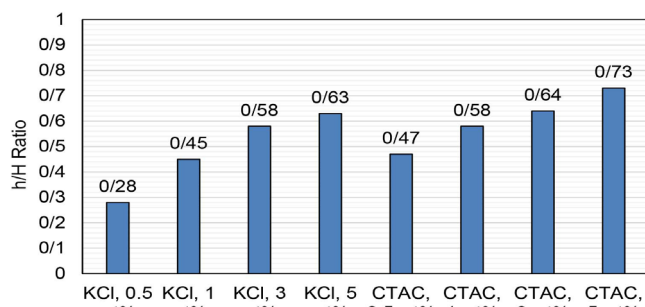


Fig. 7 Final results of the bentonite sedimentation test in the presence of CTAC and KCl.

The Results of Swelling Inhibition

Bentonite is used as a thickener and filtrate controller due to its fine and layered structure and water absorption, swelling, and hydration properties. Moreover, the inhibitor addition to the mud with a water basis reduces its capacity to hydrate, resulting in decreased rheology and its parameters, including yield point, plastic and apparent viscosities, gel strength of 10 seconds and 10 minutes, and increased filtrate. Therefore, the measurement of these parameters can describe the performance of an inhibitor. Figs. 8-12 show the results of these tests for CTAC, CTAB, and KCl. Based on the results of Figs. 8-11, the bentonite hydration is very intense without any inhibitor, and its rheological properties are higher than fluids containing an inhibitor. Also, according to Fig. 12, the filtrate removal was very low due to the formation of a thick cake. Moreover, with the addition of each CTAC, CTAB, and KCl inhibitor, the rheological properties decreased due to the weakening of the swelling. In brief, it can be concluded that the concentration of inhibitors is directly proportional to the decrease in rheological properties, increase in filtrate volume, and weak or no filter cake formation on the paper.

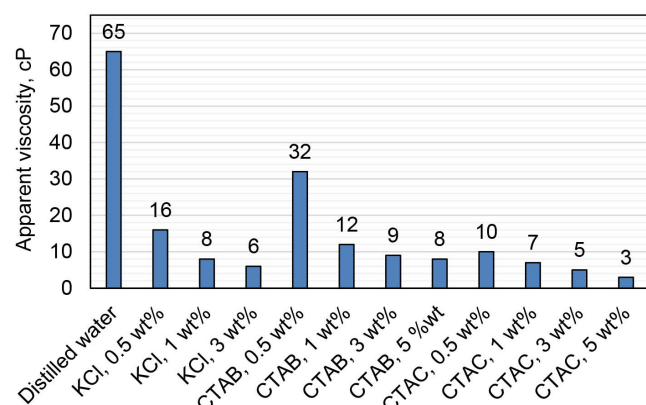


Fig. 8 Apparent viscosity values in the bentonite swelling inhibition test.

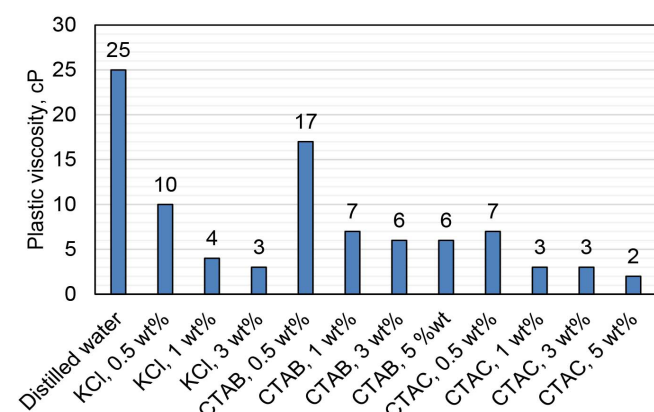


Fig. 9 Plastic viscosity values in the bentonite swelling inhibition test.

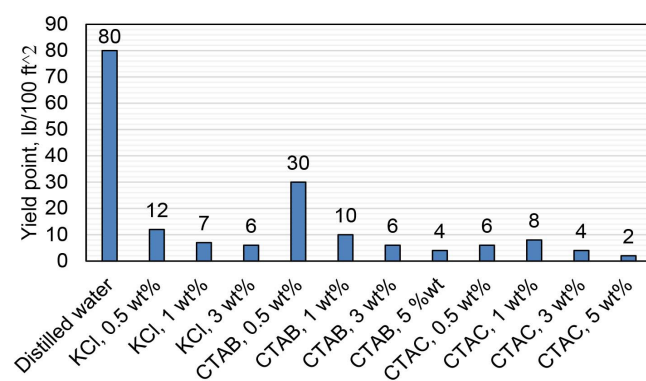


Fig. 10 Yield point values in the bentonite swelling inhibition test.

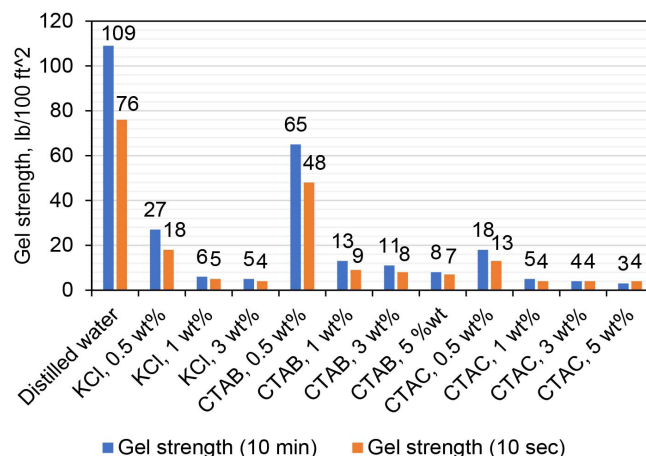


Fig. 11 Gel strength values in the bentonite swelling inhibition test.

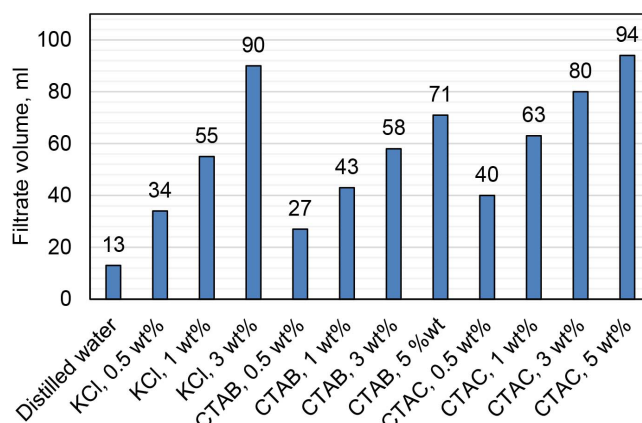


Fig. 12 Filtrate volume in the bentonite swelling inhibition test.

In a comparison of the inhibitors, it can be stated that CTAC had the best performance and CTAB had the weakest performance. The following order for the performance of the inhibitors showed:

$$\text{Distilled water} < \text{CTAB} < \text{KCl} < \text{CTAC}$$

This comparison reveals that CTAC at a lower concentration of 0.5 wt% exhibits stronger and more effective performance than CTAB at 3 wt%. Moreover, this means that CTAC is a more appropriate and efficient inhibitor than conventional CTAB. In addition, viscosity is a property of a drilling fluid that transports and suspends drilling cuttings as one of its applications.

The viscosity of aqueous-based fluids is usually reached to the desired values of bentonite. Sometimes, according to the conditions, various polymers are used as thickeners. It should be remembered that preventing the swelling of bentonite as the main purpose of inhibitors, if the inhibitor is not a polymeric, reduces bentonite's viscosity performance. Moreover, as seen, common inhibitors also reduce the viscosity of bentonite by reducing its swelling, which does not help in the suspension and transfer of the cuttings. In this case, it is possible to compensate for the decrease in viscosity by adjusting the viscosity after adjusting the concentration of the inhibitor. This also applies to cationic surfactant inhibitors. Moreover, the reasons behind the phenomena is linked to reduced interparticle interactions among clay platelets, which resulted from the adsorption of the cationic portion of surfactant molecules onto the negatively charged clay surfaces. In addition, this adsorption process effectively neutralized the negative charge through electrostatic forces [11, 38, 39].

Similarly, the filtrate increases when the inhibitor is used. This issue, while confirming the effectiveness of inhibitors, is one of their negative effects. Moreover, one of the primary objectives of drilling fluid engineering is to decrease fluid loss in drilling formulations by utilizing fluid loss additives. Moreover, numerous fluid loss additives, such as polymers and nanoparticles, have been documented in the literature for their ability to mitigate fluid loss during drilling [40, 41]. Adding shale inhibitors, such as ionic liquids and surfactants, slightly increases the fluid loss of drilling formulations. Moreover, the inhibitor molecules intercalate the clay particles by expelling the water molecules from the clay surfaces, and consequently, the fluid loss increases. Therefore, incorporating shale inhibitors like ionic liquids

and surfactants leads to a marginal rise in fluid loss within drilling formulations [11, 38, 39].

The Results of the Compatibility Evaluation

The ability of an inhibitor to work effectively with other substances added to drilling mud is essential for its effectiveness. A test was conducted on two types of drilling mud, simple and complete, to measure rheological changes and filtrate in the presence and absence of CTAC and CTAB inhibitors at 1 wt.%. Figs 13 and 14 show the results of this test. Moreover, the addition of CTAB caused more significant changes in these parameters than CTAC, indicating that CTAB is more incompatible with other additives. Conversely, CTAC exhibited acceptable compatibility with simple and complete drilling mud, as the rheological changes and filtrate were insignificant.

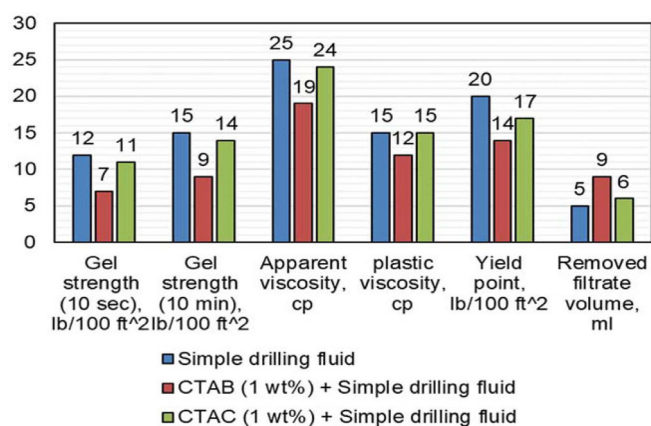


Fig. 13 Results of the simple drilling mud compatibility test.

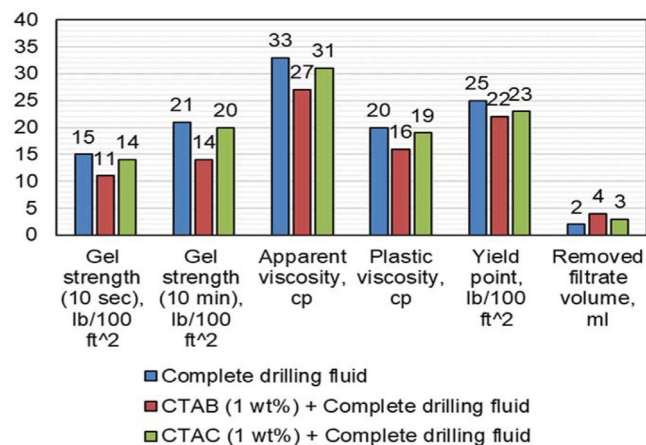


Fig. 14 Results of the complete drilling mud compatibility test.

Shale Sample Recovery

During this exam, the effects of CTAC, CTAB, and KCl inhibitors on shale recovery in both water and drilling mud environments were investigated. The inhibitors were utilized at 1 wt.% and 3 wt.%. The total and categorized recoveries when using inhibitors in a water environment are presented in Figs 15 and 16, respectively. Furthermore, the total recovery of shale from the water was found to be 20.26%, including 42.4% in the 2 mm main size and the remainder in smaller sizes. By adding CTAC, CTAB, and KCl at 1 wt.%, the total shale recovery was improved to 27.16%, 34.57%, and 39.60%, respectively.

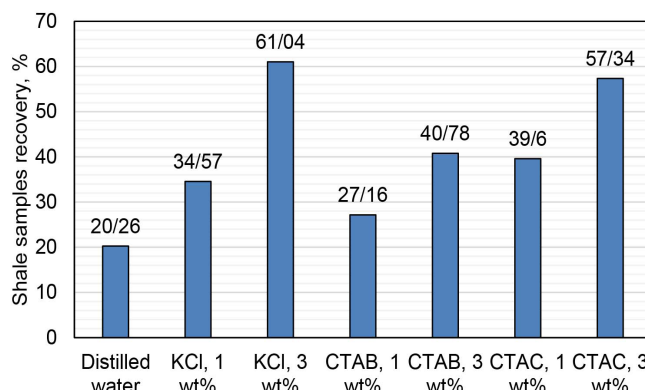


Fig. 15 Results of the total shale recovery test for all types of water environments in different concentrations of CTAC, CTAB, and KCl.

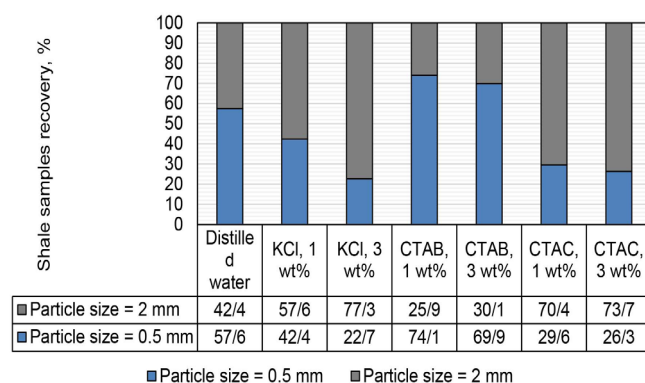


Fig. 16 Results of the categorized shale recovery test for all types of water environments in different CTAC, CTAB, and KCl concentrations.

Although CTAB yielded the highest total shale recovery, the recovered shale samples were smaller and more worn. KCl resulted in a higher total shale recovery at the original size of 2 mm. Moreover, in addition to increasing the total shale recovery by 19.34%, the recovery of shale at the original size of 2 mm was also enhanced by CTAC. This indicates the better performance of CTAC against CTAB and KCl as common inhibitors, which can be seen at the lower concentration.

The better performance of CTAC and CTAB at a lower concentration and the non-continuation of superiority at higher concentrations can be due to its micellar behavior in the drilling fluid. Moreover, the formation of micelles depends on the concentration. In general, micelles are formed spontaneously and depend on the number of agents dissolved in the environment and the concentration of surfactant in the solution. The surfactant particles are dispersed separately in the solution when the surfactant concentration is less than a certain limit. Increasing the surfactant concentration to a certain level causes micelles to form, and this process continues gradually. Moreover, this concentration limit at which micelles begin to form is known as the critical micelle concentration (CMC). After the formation of micelles, the activity of surfactants in the limited environment and their performance is weakened [42, 43]. In addition, this can be the reason for the sudden change in the rheological behavior with the increase in the concentration of surfactants in the previous part.

Shale recovery is proportional to the concentration of inhibitors and increases with increasing concentration. At a concentration of 3 wt.%, CTAB obtained the lowest and CTAC the highest shale recovery, respectively. In addition, CTAC recovered shale with larger original sizes. In other words, in addition to the highest shale recovery, the highest virgin and original-size samples were also obtained using CTAC. The recovery of virgin samples did not show significant changes despite the increase in the concentration of CTAB and KCl, which resulted in increased shale recovery. In other words, these two inhibitors act weaker than CTAC in shale stability against water penetration.

Fig. 17 and 18 show the results of CTAC, CTAB, and KCl stabilizers on shale recovery in different drilling muds. The total shale recovery of simple and complete drilling muds without inhibitors was 62.11% and 89.57%, respectively. The recovery of samples with the original size for simple and complete muds was equal to 44.9% and 67.1% of the total recovered samples, respectively. By adding CTAB, total shale recoveries in simple and complete drilling muds increased by 5.53% and 0.94%, respectively. Meanwhile, CTAC showed the best performance in this test. In the presence of CTAC, the increase in total recoveries in simple and complete drilling muds was 12.37% and 6.43%, respectively.

On the other hand, CTAB did not have much effect on the total shale recovery in each sample of drilling mud, and a slight increase in recovery was recorded. Furthermore, the recovery of samples in their original size in both types of drilling mud decreased with the addition of CTAB, indicating that CTAB caused more shale erosion. This is in contrast to the performance of CTAC, which not only leads to a rise in the total recovery of shale but also results in less erosion and better recovery of samples in the main size category.

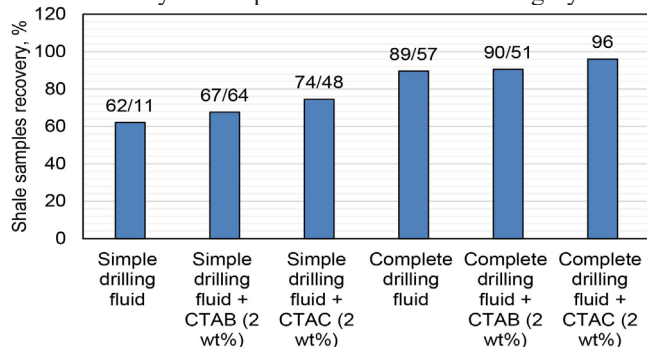


Fig. 17 Results of the total shale recovery test for all types of drilling mud in the presence of CTAC and CTAB in different concentrations.

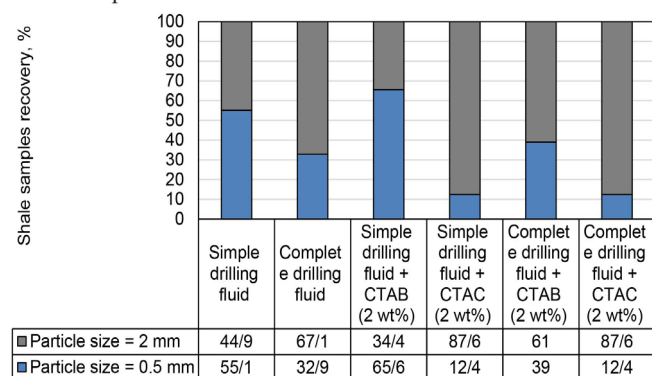


Fig. 18 Results of the categorized shale recovery test for all types of drilling mud in the presence of CTAC and CTAB in different concentrations.

Dynamic Linear Swelling

This test investigates and compares the CTAC, CTAB, and KCl effects on tablet swelling in water environments and simple and complete drilling muds. Furthermore, the experiments were conducted at 25 °C for water environments and 75 °C for drilling mud. Moreover, the results of the tests are presented in Figs 19-22. The swelling behavior was observed to follow a similar pattern, increasing significantly at the beginning. This trend was similar to the sodium montmorillonite swelling observed in previous studies [44]. The bentonite swelling in water environments with various concentrations of CTAC is illustrated in Fig. 19.

Moreover, at zero concentration, the bentonite tablet swelled continuously from the beginning of the experiment until it reached 119% swelling at the end after 3000 min. The tablet swelling was significantly affected by CTAC, which showed a good performance at low concentrations and reduced swelling compared to water. The presence of CTAC at 0.5 wt% resulted in a 36% reduction in swelling. Increasing the inhibitor concentration led to a greater swelling reduction, with a 72% reduction observed at the end of the experiment at 3 wt%. However, when the CTAC amount reached 0.5 wt%, no significant reduction in swelling was observed. This means there was an optimal performance concentration of 3 wt% for CTAC.

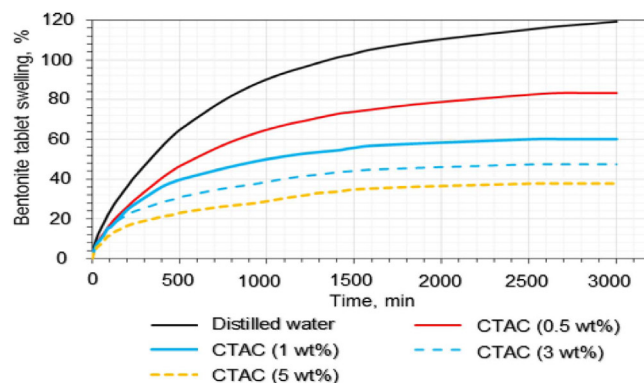


Fig. 19 The results of the dynamic linear swelling test of bentonite in the presence of CTAC in different concentrations at 25 °C.

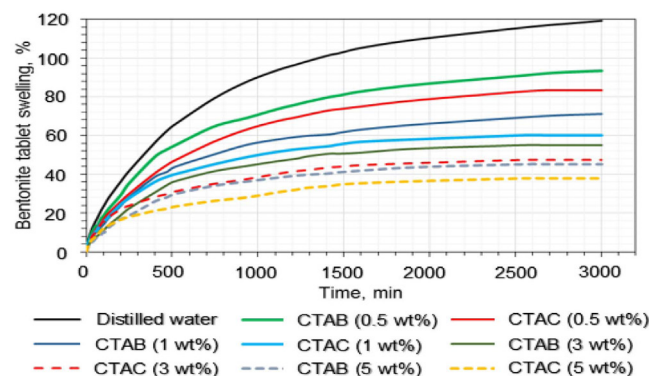


Fig. 20 The results of the dynamic linear swelling test of bentonite in the presence of CTAC and CTAB at different concentrations and 25 °C.

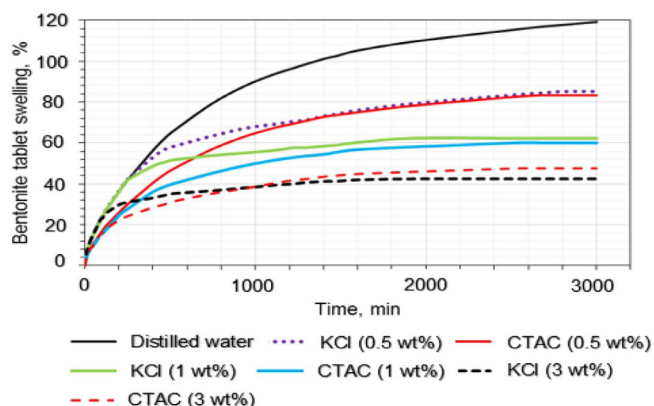


Fig. 21 The results of the dynamic linear swelling test of bentonite in the presence of CTAC and KCl at different concentrations at 25 °C.

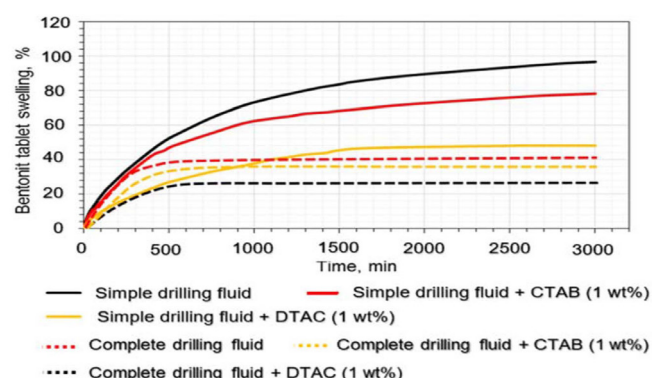


Fig. 22 The results of the dynamic linear swelling test of bentonite in the medium of simple and complete drilling mud containing CTAC and CTAB in a concentration of 1 wt% at 70 °C.

It can be seen that the presence of the inhibitor caused the tablet swelling to stop before the full duration of the experiment, which was 3000 minutes. Moreover, this means that increasing the concentration of the inhibitor reduced the bentonite swelling against water and shortened the swelling time. In addition, specifically, at a concentration of 0.5 wt%, the tablet swelling reached its maximum after 2500 minutes, and no further change in volume was seen. At 5 wt%, changes in the volume of the bentonite tablet stopped after 1500 minutes. The tablet swelling in the CTAC, CTAB, and KCl aqueous mediums are shown in Figs 20 and 21. In Figure 20, it is demonstrated that CTAC outperformed CTAB in managing bentonite swelling. The swelling rate of the bentonite tablet exposed to a surrounding containing 5 wt% CTAB was nearly equivalent to the swelling rate of the tablet in touch with an environment containing 3 wt% CTAC.

Fig. 21 illustrates the comparison between the effects of CTAC and KCl on reducing tablet swelling. Overall, the performance of these two inhibitors was similar in terms of their ability to prevent the tablets from swelling in water. Both were effective in delaying the swelling and collapse of shale samples, which is crucial in reducing the instability of the wellbore while drilling. Moreover, the findings indicate that the bentonite swelling rate is initially high in the presence of KCl, similar to that in distilled water. Still, as time progresses, the rate reduces and deviates from the distilled water trend. In contrast, the presence of CTAC effectively controls and reduces the swelling of the bentonite tablet from the beginning of the experiment. Moreover, the ineffectiveness

of KCl in controlling the swelling of the bentonite tablet is apparent from the early stages, which could lead to problems and, ultimately, instability of the well wall during drilling. In another part, the performance of CTAC and CTAB was investigated in the dynamic linear swelling test in different drilling muds (with the composition given in Table 1) at 70 °C. Fig. 22 shows the results of these tests for simple and complete drilling muds at a concentration of 1 wt.% of the inhibitors. The bentonite tablet swelled greatly in simple drilling mud without inhibitors, so after 3000 min, its swelling reached 96.6%. Furthermore, even after this time, the swelling did not stop. Adding CTAB to simple drilling mud decreased the swelling rate by 18.5%. The swelling reduction by adding CTAC to simple drilling mud reached 42.7%. This shows that CTAC performs better than CTAB. Moreover, the bentonite swelling with CTAC was observed to stop earlier than in the presence of CTAB. In addition, specifically, the swelling stopped at 1700 min with CTAC, whereas it persisted until the completion of the examination with CTAB. However, the inhibitory effects of both CTAC and CTAB were observed from the initial stages of the test. The bentonite swelling in complete drilling mud is shown in Fig. 22. The bentonite swelling rate in complete drilling mud is less than in simple drilling mud due to inhibitors such as PHPA, NaCl, and KCl in the complete mud composition. Moreover, in the end, the bentonite swelling in the complete drilling mud was 40%. The addition of CTAC and CTAB in the complete drilling mud reduced the swelling by 9.94% and 4.2%, respectively, indicating that CTAC is more effective than CTAB. In addition, the swelling of bentonite tablets in complete drilling mud stops in a shorter time than in simple drilling mud due to the presence of inhibitors.

The Results of Mechanistic Experiments

Wettability Test

The wettability analysis is shown in Fig. 23. The water droplet was completely adsorbed on the bentonite-coated glass slide, and the contact angle was 25°, as seen in Fig. 23a. Water absorption by bentonite can be justified due to its polarity and negative charge of bentonite sheets. For this reason, bentonite has a very hydrophilic wettability. In the presence of KCl in the bentonite mixture attached to the glass slide, the water drop was quickly adsorbed and formed a contact angle of 38°. As seen in Fig. 23b, this indicates the preservation of the initial hydrophilicity wettability of bentonite. Moreover, the adsorption of the water droplet on the bentonite-coated glass slide containing CTAB was weak, indicating the wettability alteration with a contact angle of 77° (Fig. 23c). In addition, the hydrophobic state became more intense in the presence of CTAC, and the water drop on the glass slide was stable, having a contact angle of 75°, as seen in Fig. 23d.

Zeta Potential

The zeta potential for a bentonite suspension with the CTAC inhibitor is shown in Fig. 24. Zeta potential measures the suspension's stability and the electrostatic forces' intensity among dispersed clay particles. Moreover, a higher zeta potential value shows that the clay particles are more stable in the water environment.

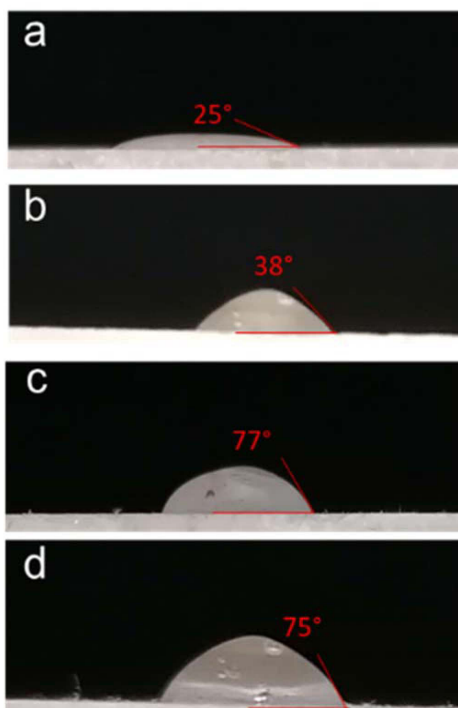


Fig. 23 The results of bentonite wettability test: a) distilled water, b) KCl, c) CTAB, d) CTAC.

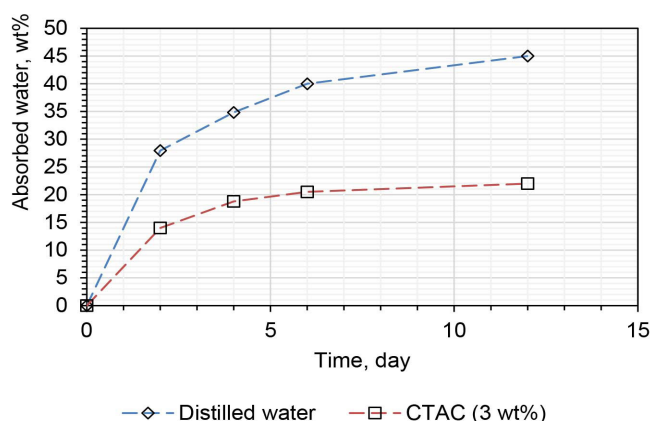


Fig. 24 Results of the zeta potential test of suspension containing pure bentonite and CTAC in different concentrations.

When clay particles aggregate and clump together, the zeta potential decreases. In addition, the zeta reduction indicates that the clay particles are less responsive to water [45].

The results of Fig. 24 show that initially, the suspension has a zeta potential of -39.5 mV, indicating high stability, as values greater than ± 30 mV typically signify good stability due to strong repulsive forces between particles preventing aggregation. However, when CTAC, a surfactant, is added at various concentrations (0.5, 1, 3, and 5 wt.%), the zeta potential decreases to -28.9 mV, -26.9 mV, -21.2 mV, and -17 mV, respectively.

This reduction in zeta potential suggests a corresponding decrease in suspension stability as the particles become less charged and more prone to aggregation. The decrease in stability with increasing CTAC concentration is attributed to the neutralization of surface charges on the particles, leading to reduced repulsive forces and an increased likelihood of particle aggregation.

The Isothermal Water Absorption

The outcomes of the isothermal water absorption experiment are demonstrated in Fig. 25. In the first 2 days, strong water absorption was evident for both samples. However, water absorption was much lower in the presence of the inhibitor. Moreover, the pure bentonite absorbed water until the end of 12 days, but the inhibitor-modified bentonite did not absorb much water after 6 days. In addition, this reduction in water absorption is a sign of the inhibitor's performance in shale stability. Furthermore, reduced water absorption will result in less swelling and disintegration.

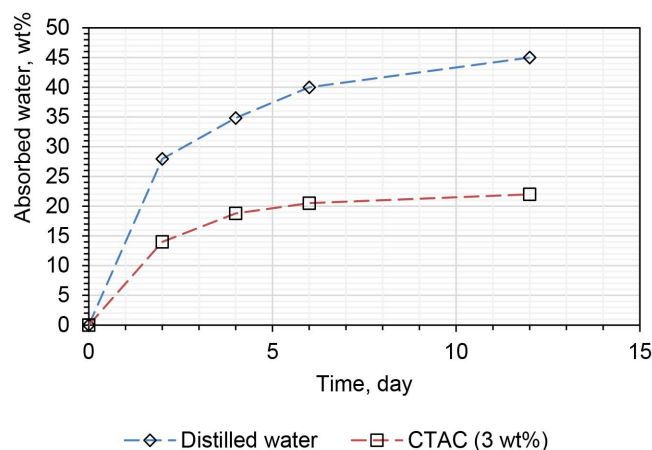


Fig. 25 Results of the isotherm water absorption test of pure bentonite and bentonite modified with CTAC.

The results of Thermal Gravimetric Analysis

TGA analysis was performed on pure and modified bentonite samples with CTAC, CTAB, and KCl inhibitors and compared. The results of this analysis are shown in Fig. 26. A similar trend can be observed for all samples. TGA revealed three distinct weight loss steps across all samples, corresponding to different temperature ranges: below 200 °C, between 200 °C and 500 °C, and above 500 °C.

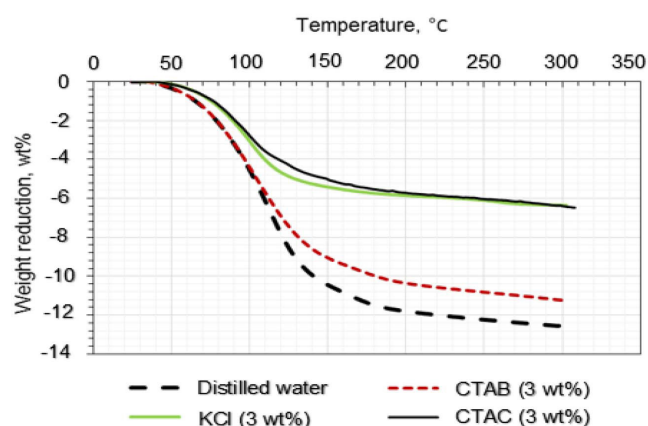


Fig. 26 TGA analysis results of bentonite samples modified with CTAC, CTAB, and KCl at a concentration of 3 wt%.

The results of Thermal Gravimetric Analysis

TGA analysis was performed on pure and modified bentonite samples with CTAC, CTAB, and KCl inhibitors and compared. The results of this analysis are shown in Fig. 26. A similar trend can be observed for all samples. TGA revealed three distinct weight loss steps across all samples,

corresponding to different temperature ranges: below 200 °C, between 200 °C and 500 °C, and above 500 °C.

The initial weight loss, occurring at temperatures below 200 °C, is primarily attributed to the evaporation of water. This includes the physically adsorbed water on the surface of the bentonite plates and the water molecules associated with the interlayer cations, which are loosely bound within the clay structure. This step is critical, as it reflects the bentonite's moisture content and hydration state [45].

The second weight-loss phase, occurring between 200 °C and 500 °C, is associated with the decomposition of organic materials within the bentonite matrix. Moreover, these organics, often residual from natural sources or introduced during processing, undergo thermal degradation, leading to a noticeable mass reduction. Furthermore, this step highlights the presence of organic contaminants or additives and the thermal stability of the bentonite [46].

The third and final weight loss stage, observed at temperatures above 500 °C, corresponds to the dehydroxylation of the bentonite structure. This involves the loss of hydroxyl groups from the octahedral sheets of the bentonite, which is a crucial process in understanding the clay's thermal stability and structural integrity.

Dehydroxylation typically leads to significant alterations in the clay's structure, potentially affecting its physical and chemical properties [37].

Quantitatively, pure bentonite exhibited the most substantial overall weight loss, totaling 12.24%. Moreover, this significant reduction highlights the high moisture content and organic and hydroxyl groups within the pure clay. In contrast, bentonite modified with cetyltrimethylammonium bromide (CTAB) at a concentration of 3 wt.% demonstrated a lower total weight loss of 10.83%.

The reduced weight loss in the modified sample suggests that the CTAB modification enhances the thermal stability of the bentonite, likely by altering the surface characteristics and reducing the availability of water and organic materials within the clay structure. Moreover, the modification of bentonite with CTAC at 3 wt.% resulted in the most significant reduction of the intra-network water in bentonite. The weight losses showed that bentonite treated with KCl had a final weight loss of 6.1%, while bentonite modified by CTAC had a final weight loss of 6.03%. In addition, this indicates that CTAC has a stronger performance compared to common inhibitors such as CTAB and KCl. This behavior of CTAC can be compared with the behavior of the similar surfactant of Dodecyltrimethylammonium chloride because when bentonite was modified by reacting with it at a concentration of 3 wt%, the weight loss was almost equal to the one of CTAC, and the same trend in Fig. 26 against temperature was observed [11].

Possible Mechanism of the CTAC Activity

After conducting various tests and analyzing the chemical structure of CTAC, we propose a possible mechanism in this section. Bentonite, a smectite clay mineral, has a layered structure where each layer comprises an octagonal alumina sheet sandwiched between two hexagonal silica sheets. In an aqueous medium, water molecules with a polar nature are adsorbed to the oxygen atoms on the silica surface through

hydrogen bonds when the bentonite particles come in contact with water [47, 48]. Furthermore, when other hydrogen bonds replace hydrogen bonds between the water molecules and the outer silica plate surface, the water adsorption on these plates decreases and is delayed. Moreover, as a result, the bentonite hydration and swelling decrease. Glycols, their derivatives, various amines, and amines-based, utilized as shale stabilizers in drilling fluids, get adsorbed on the bentonite sheets surface by forming hydrogen bonds [37, 49, 50].

By replacing water molecules with CTAC on the bentonite, the adsorption of water molecules is reduced, leading to a decrease in the swelling of bentonite particles. CTAC has a nitrogen atom in the center linked to four carbon atoms through covalent bonds. This charged nitrogen atom can bind to the negatively charged surface of bentonite plates, thus becoming trapped between the layers of bentonite particles. The amine's nitrogen atoms can remove primary cations in the layers of bentonite particles by bonding to them, causing intercalation (see Fig. 27a).

CTAC and water competition for adsorption on the bentonite limits the available sites for water adsorption and reduces the bentonite hydration and swelling. Additionally, the adsorption of CTAC and CTAB molecules alters the wettability of the surface of the plates and increases their hydrophobicity, according to the wettability test results. The alkyl chain attached to the central nitrogen atom in the amine accounts for this capability (see Fig. 27b).

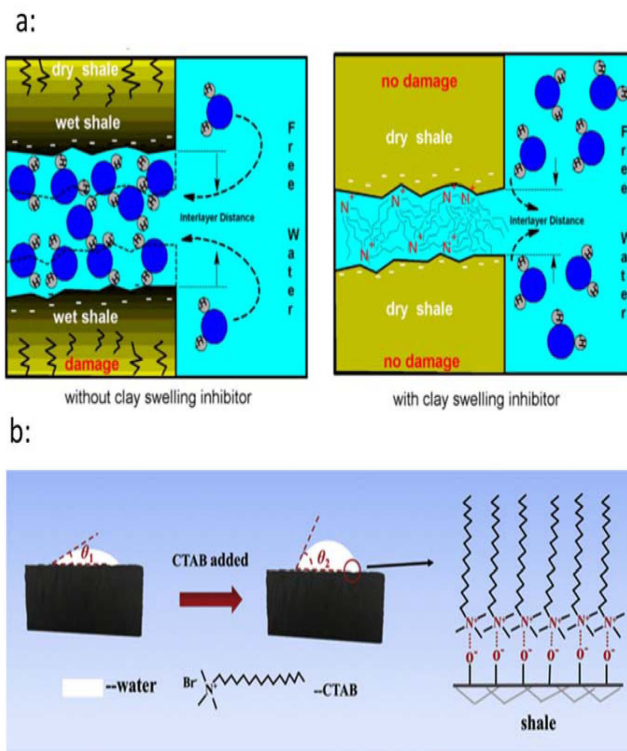


Fig. 27 a) The swelling inhibition mechanism by cationic inhibitors [51] and b) Wettability alteration by cationic surfactant [52].

Conclusions

The swelling inhibition properties of CTAC and its mechanism of action were investigated by performing tests and comparing them with two common inhibitors. Moreover, the results confirm the appropriate performance of CTAC as a new inhibitor. In addition, the results are briefly sorted below:

- CTAC is compatible with other drilling mud additives and does not weaken their performance.
- This inhibitor has a great effect on reducing swelling and increasing the stability of bentonite.
- By adsorbing CTAC on the bentonite surface, it altered the wettability to hydrophobicity, which reduces water absorption by bentonite as the contact angle of the water drop on the glass slide was 75° and while the initial contact angle in the absence of CTAC was 25°.
- CTAC performed better and stronger than the common inhibitors CTAB and KCl in preventing water absorption and stabilizing bentonite. Moreover, the presence of CTAC at 0.5 wt.% resulted in a 36% reduction in swelling. In addition, increasing the inhibitor concentration led to a greater swelling reduction, with a 72% reduction observed at the end of the experiment at 3 wt%. However, when the CTAC amount reached 0.5 wt.%, no significant reduction in swelling was observed.
- When CTAC molecules are adsorbed on the bentonite particles, their surfaces lose the intensity of negative charges, reducing the clay sensitivity to water molecule absorption, as demonstrated in the isothermal water absorption test. As a result, the mineral stability increases.
- Ultimately, based on the TGA analysis, CTAC molecules penetrate the interlayer configuration of clay sheets, decreasing the presence of water molecules in the network.

Abbreviations

AV: Apparent viscosity
 CMC: Critical micelle concentration
 cP: Centipoise
 CTAB: Cetyltrimethylammonium bromide
 CTAC: (Carboxymethyl)trimethylammonium chloride
 g/mol: Grams per mole
 HV: High viscosity
 KCl: Potassium chloride
 ml: Milliliter
 MPa: Megapascal
 mV: Millivolt
 NaCl: Sodium chloride
 NaOH: Sodium hydroxide
 Pa: Pascal
 PAC-LV: Low viscosity polyanionic cellulose
 PAC-R: Regular viscosity polyanionic cellulose
 PHPA: Partially hydrolyzed polyacrylamide
 psi: Pounds per square inch
 PV: Plastic viscosity
 rpm: Revolutions per minute
 TGA: Thermogravimetric analysis
 wt%: Weight percent
 XRD: X-ray diffraction

YP: Yield point

Authors Declaration

The authors have no conflicts to disclose.

Data will be available on request.

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