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Investigation Of Wettability Alteration By Clay Stabilizer

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ALTERATION BY CLAY STABILIZER

Investigation Of Wettability Alteration By Clay Stabilizer

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DECLARATION OF DISSERTATION

Title of thesis: **Investigation Of Wettability Alteration By Clay Stabilizer**

I MAHMOUD KHALED MOSTAFA ABBAS MOHAMED

hereby declare that the dissertation is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UTP or other institutions.

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ABSTRACT

Wettability is a pivotal property influencing a range of industrial activities, notably in oil recovery and surface coatings. Clay stabilizers, encompassing Cetyltrimethylammonium bromide (CTAB) and Polydiallyldimethylammonium chloride (PolyDADMAC), serve vital roles in drilling operations, particularly in circumventing fluid loss and safeguarding wellbore stability. This research delved deeply into understanding how these stabilizers impact wettability and their mechanism in averting clay swelling.

Through rigorous experimental investigations, the study shed light on the intricate effects of various clay stabilizers on wettability shifts. Their compatibility across diverse salinities was evaluated, and the consequent influence of their concentrations on the contact angle of a test liquid was meticulously observed. The KRUSS Drop Shape Analyser was pivotal in gauging contact angles, while advanced analytical techniques like FTIR, SEM, and EDS granted nuanced insights into material interactions and morphological alterations.

The amassed results offer a detailed understanding of the functionality of clay stabilizers across different environments, elucidating the nuanced mechanisms leading to wettability changes. This exploration not only enhances the comprehension of the interrelationship between wettability and clay stabilization but also carries profound implications for sectors spanning oil and gas to specialized coatings. Conclusively, the discoveries made in this research are paramount, potentially steering significant enhancements in diverse industrial applications and adding a valuable chapter to material science literature.

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INTRODUCTION

1.1 Background Study

Wettability is a fundamental property of materials, dictating their propensity to spread and adhere to a solid interface. Its pivotal role spans various sectors, notably oil recovery, underscoring its significance. To fortify wellbore stability and curtail fluid loss during drilling operations, clay stabilizers, such as Cetyltrimethylammonium bromide (CTAB) and Polydiallyldimethylammonium chloride (PolyDADMAC), have been routinely integrated into drilling fluid compositions. The alteration in wettability encompasses the transition of a solid surface's affinity between fluids, predominantly water and oil. In the realm of curtailing clay swelling, the principal objective was transitioning the clay's wettability from water-wet to oil-wet, or towards an intermediate wettability state.

The aim of this research was to elucidate the influence of clay stabilizers on solid surface wettability. Evaluations were conducted to discern the impact of diverse stabilizers and their concentrations on the contact angle of a test liquid, granting insights into wettability modification mechanisms. The findings from this research could potentially steer advancements in myriad industrial procedures, accentuating its invaluable contribution to material science.

When exposed to fluids of low salinity or elevated pH, clays can undergo swelling and facilitate the migration of fine particles. Such interactions mandate the incorporation of clay stabilizers in multifaceted treatments. Upon interaction with low-salinity fluids, the detachment of clay particles can ensue, and if these dispersed entities lodge within pore throats, a decline in permeability might be observed. Given that clays can absorb water, inflating to nearly 20 times their initial volume, the swelling is typically deterred using an array of clay stabilizers. Their modus operandi entails the encapsulation of clay's active facets, thus repelling the water phase and consequently curbing clay swelling.

In reservoir operations, clay stabilizers have been ubiquitously employed. A plethora of clay stabilizers exist, each exhibiting distinct operational mechanisms and efficacies. Prominently, quaternary ammonium compounds (QACs) function by encasing active clay particle surfaces, repulsing the water phase, and consequently thwarting clay swelling. Such QAC-centric stabilizers have manifested efficacy in curbing clay swelling tendencies in low-salinity environments. Another variant, rooted in polyamides, operates by occluding active clay surface sites, precluding water interaction. Particularly in environments marked by elevated temperatures and pressures, stabilizers like KCl, CTAB, and PolyDADMAC demonstrated superior performance. Numerous other stabilizers, including those based on phosphonates and amino acids, have been developed and assessed for their prowess in clay swelling and migration prevention. However, the performance spectrum of these stabilizers can oscillate based on reservoir-specific conditions such as salinity, pH, and temperature.

In this research, a comprehensive experimental exploration was executed to gauge the effects of diverse chemicals, assessing their compatibility across varying salinities. The overarching objective was discerning how stabilizer concentrations modulate solid surface wettability and the subsequent alteration mechanisms.

1.2 Problem Statement

Clays can cause swelling and fine particle migration when they come into contact with low salinity or high pH fluids, reducing permeability and leading to better understanding of the Reservoir conditions. However, the interaction between the clay stabilizer and the surrounding fluids, as well as the concentration of the stabilizer, can impact its ability to prevent clay swelling and migration.

The current understanding of the mechanism of wettability alteration by clay stabilizers is limited low salinity and clay composition , and the compatibility of the stabilizer at different salinity levels and its effect on wettability alteration is not well understood. There is a need for research to improve the understanding of the relationship between wettability and clay stabilizers, and to optimize their use in industrial applications.

The presence of clays in reservoirs can lead to issues such as swelling, fines migration, and reduced permeability, all of which can have a significant understanding of the reservoir conditions. To mitigate these issues, clay stabilizers are often used in drilling fluid formulations. However, the effectiveness of clay stabilizers can be impacted by factors such as the concentration of the stabilizer and its compatibility with different salinity levels. In addition, the mechanisms by which clay stabilizers alter wettability are not well understood. From a reservoir engineering perspective, it is crucial to understand how clay stabilizers interact with surrounding fluids and how they impact wettability alteration. By investigating the effect of varying concentrations of clay stabilizers and their compatibility with different salinity levels, we can gain a better understanding of the relationship between clay stabilization and wettability alteration.

Therefore, this research aims to investigate the effect of varying concentrations of clay stabilizers on the alteration of wettability and assess the compatibility of the stabilizer at different salinity levels to provide insights into the optimization of the use of clay stabilizers in industrial applications.

1.3 Project Objectives

1- To evaluate the compatibility of the clay stabilizer with different levels of salinity to determine its efficacy in preventing clay swelling and migration in varying conditions.

2- To investigate the impact of varying concentrations of clay stabilizers on the alteration of wettability of solid surfaces. Through this research, the relationship between clay stabilizer concentration and wettability alteration will be explored to optimize the use of clay stabilizers and enhance their capability to prevent clay swelling.

1.4 Study Scope

The scope of the research on "Wettability Alteration by Clay Stabilizer" delves deeply into the compatibility and interaction of select clay stabilizers under diverse salinity levels of brine and distinct environmental parameters. The solutions explored involve KCl, PDADMAC, and CTAB, combined with brine and 0.6g Bentonite. These will be studied under brine salinity concentrations of 1.5 wt%, 3.5 wt%, and 5.5 wt%. Moreover, the research will assess the performance of these stabilizers under two distinct temperature conditions: room temperature and 86.6°C, and at two pressure settings: room pressure and 1450 PSI. By doing so, the study aims to offer a profound understanding of wettability alteration by these clay stabilizers, underlining their potential and relevance in diverse industrial contexts.

Table 1 Study Scope

Parameter	Description
Salinity Levels	1.5wt%, 3.5wt%, 5.5wt%
Temperature	24°C , 86.6°C
Pressure	14.6 PSI , 1450 PSI
Type of Rock	Sandstone
Clay Stabilizers	KCl,Polydiallyldimethylammoniumchloride(PDADMAC), Cetyltrimethylammoniumbromide (CTAB)
Stabilizer Concentration	0.2wt%, 0.8wt%, 1.4wt%, 2.0wt%

CHAPTER 2

LITRETURE REVIEW

2.1 Introduction to Wettability

Wettability is a crucial property of materials that determines their ability to spread and adhere to a solid surface (Goddard, 2002). It plays a critical role in several industrial processes, including oil recovery, surface coating, and printing, making it an important area of study et al. Zhao et al. (2019). Wettability influences the wetting, spreading, and adhesion behavior of liquids on solid surfaces and has significant implications (Rafiee, 2016).

In the oil and gas industry, wettability plays a vital role in determining the efficiency of oil recovery (Bai, Liu, 2017). The contact angle between the oil and rock surface affects the capillary forces that control the movement of fluids through the reservoir (Zhang, 2019). If the oil is strongly adhered to the rock surface, it becomes difficult to displace, and recovery rates are low. However, if the oil is weakly adhered to the rock surface, it can be more easily displaced, leading to higher recovery rates (Gubaidullin, 2017).

In summary, wettability is a critical property that plays a significant role in various industrial processes, including oil recovery. Understanding the mechanisms of wettability and developing strategies to modify it is crucial for improving the Reservoir and lab values.

2.2 Contact Angle and Wettability

In any scientific investigation concerning surface interactions, understanding the concept of contact angle and its relationship to wettability is crucial. The contact angle refers to the angle formed at the three-phase boundary where a liquid, gas, and solid meet. It's a measure of the wettability of a surface, a critical property that influences how a liquid will spread or bead up on a surface. In this study, we observed significant variations in contact angles across our experimental groups, signifying differences in the wettability of the glass surface with the introduction of different additives.

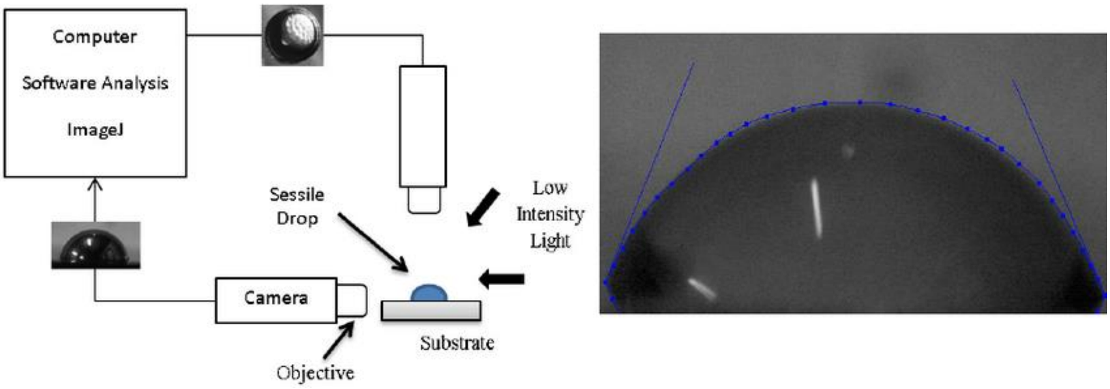


Figure 1 Contact Angle Goniometer

2.3 Types of clay stabilizers

Clay stabilizers are commonly used in reservoir evaluating. When clay minerals come into contact with low-salinity or high-pH fluids, they can cause swelling and fine particle migration. This, in turn, can reduce the permeability of the formation. To prevent this from happening, different types of clay stabilizers have been developed and tested for their effectiveness in inhibiting clay swelling and migration.

In the oil and gas industry, clay stabilizers are essential in preventing clay swelling, which can cause formation damage, reduce production rates, and complicate drilling operations. Different types of clay stabilizers are used, including salt-based, polymer-based, and surfactant-based stabilizers. Here, we discuss the performance of these stabilizers, specifically KCl (potassium chloride), CTAB (cetyltrimethylammonium bromide), and PolyDADMAC (poly(diallyldimethylammonium chloride)) under different conditions.

One common type of clay stabilizer is based on quaternary ammonium compounds (QACs), which work by coating the active surfaces of clay particles and repelling the water phase, thereby hindering clay swelling. QAC-based clay stabilizers have been found to be effective in reducing the tendency of clays to swell and migrate in low-salinity fluids (Ghaderi et al., 2015). Another type of clay stabilizer is based on polyamides, which work by blocking the active sites on clay surfaces, thereby preventing water from interacting with them. Polyamide-based clay stabilizers have been found to be particularly effective in high-temperature and high-pressure environments (Zhao et al., 2014). Other types of clay stabilizers, such as phosphonate-based and amino-acid-based stabilizers, have also been developed and evaluated for their effectiveness in preventing clay swelling and migration.

The performance of each type of clay stabilizer can vary depending on the specific conditions, such as salinity, pH, and temperature. In a study conducted by Liu et al. (2019), the performance of different types of clay stabilizers was evaluated in different salinity conditions. The results showed that QAC-based stabilizers were more effective in low-salinity environments, while polyamide-based stabilizers were more effective in high-salinity environments.

In conclusion, the use of clay stabilizers is crucial in Reservoir evaluating. Different types of clay stabilizers have been developed and tested for their effectiveness in inhibiting clay swelling and migration. The effectiveness of clay stabilizers in preventing fluid loss has been demonstrated in several studies, both in laboratory and field settings.

2.4 Effect of Clay Stabilizers on Wettability of Solid Surfaces

Wettability is a crucial property of materials that affects their ability to spread and adhere to a solid surface.

Clay particles are known to have hydrophilic surfaces, which means they have a natural affinity for water molecules. This affinity for water can lead to swelling and migration of clay particles, resulting in decreased permeability. Clay stabilizers work by altering the wettability of clay particles, making them less hydrophilic and more hydrophobic. This change in wettability reduces the affinity of clay particles for water molecules, thus preventing swelling and migration.

The mechanism of wettability alteration by clay stabilizers varies depending on the type of stabilizer used. QACs work by adsorbing onto the surface of clay particles, forming a hydrophobic barrier that repels water molecules. This mechanism of action has been demonstrated in several studies. For example, a study by Letaief et al. (2017) evaluated the performance of QAC-based clay stabilizers in inhibiting swelling and migration of clay particles. The study found that QAC-based stabilizers effectively reduced the tendency of clays to swell and migrate in low-salinity fluids, leading to improved wellbore stability.

Polyamides are another type of clay stabilizer. Unlike QACs, polyamides work by blocking the active sites on clay surfaces, preventing water molecules from interacting with them. This mechanism of action has been demonstrated in several studies. For example, a study by Torsæter and Åkra (2004) evaluated the performance of polyamide-based clay stabilizers in inhibiting clay swelling and migration in high-temperature and high-pressure environments. The study found that polyamide-based stabilizers were particularly effective in these harsh conditions.

In addition to QACs and polyamides, other types of clay stabilizers have been developed and evaluated for their effectiveness in inhibiting clay swelling and migration. For example, phosphonate-based and amino-acid-based stabilizers have shown promise in some studies. However, the performance of each type of stabilizer can vary depending on the specific conditions of the Reservoir.

In conclusion, the wettability of solid surfaces is curtail as the effectiveness of clay stabilizers depends on their ability to alter the wettability of clay particles. Different types of clay stabilizers have different mechanisms of action, such as adsorption and surface blocking, Adsorption is a surface phenomenon where molecules or ions from a fluid adhere to the surface of a solid material, altering its wettability and chemical properties. Surface blocking is the process where adsorbed molecules or particles form a barrier on the solid surface, preventing interaction with other substances, such as water molecules, and minimizing swelling or other undesirable reactions. which can affect their performance in different conditions. A better understanding of the effect of clay stabilizers on wettability can lead to improved product quality and process efficiency in various industries.

2.5 Mechanism of wettability alteration by clay stabilizers

The mechanism of wettability alteration by clay stabilizers is a critical aspect to understand in the field of material science (Huang et al., 2019). Coating the active surfaces of clay particles is one of the primary mechanisms of wettability alteration by clay stabilizers, as it repels the water phase, hindering clay swelling and preventing permeability reduction that can occur when dispersed clay particles become trapped in pore throats (Kashefi et al., 2018). Another mechanism of wettability alteration by clay stabilizers is blocking the active sites on clay surfaces. Polyamide-based clay stabilizers, for example, work by blocking the active sites on clay surfaces, thereby preventing water from interacting with them (Jia et al., 2020). However, overusing clay stabilizer molecules can lead to unnecessary accumulation, increased costs, waste of materials, and potential negative environmental impacts, making it crucial to optimize the amount of stabilizer used for effective wettability alteration (Kashefi et al., 2018).

The performance of clay stabilizers can be affected by various factors, such as the salinity and pH of the fluid (Jia et al., 2020). For instance, QAC-based clay stabilizers have been found to be effective in reducing the tendency of clays to swell and migrate in low-salinity fluids but lose effectiveness as the salinity of the fluid increases (Kashefi et al., 2018). Similarly, the effectiveness of polyamide-based stabilizers decreases in low-pH environments (Huang et al., 2019). Therefore, understanding the factors that affect the performance of clay stabilizers is critical in choosing the appropriate stabilizer for specific applications.

2.6 Swelling and migration of clays and the need for stabilizers

Clay minerals are ubiquitous in subsurface formations and can cause a range of problems for industrial processes. One of the most significant issues is clay swelling, which occurs when clays absorb water and expand in volume, often leading to reduced permeability and fluid loss. Clays can also migrate and clog pore throats, reducing the efficiency of oil recovery (Kumar et al., 2015).

Clay stabilizers are chemical compounds that can inhibit the swelling and migration of clays by modifying their surface properties. The mechanism of action depends on the type of stabilizer, but it typically involves coating the clay particles and repelling water, which prevents the swelling and migration of clay particles (Xu et al., 2016). There are several types of clay stabilizers available, including quaternary ammonium compounds (QACs), polyamides, phosphonates, and amino-acid-based stabilizers (Babakhani & Jafari, 2019).

The effectiveness of clay stabilizers depends on several factors, including the type and concentration of the stabilizer, the salinity and pH of the fluid, and the temperature and Reservoir pressure (Savari et al., 2018). In low-salinity fluids, clays tend to absorb water and expand, which makes QACs the most effective stabilizer. However, in high-salinity fluids, QACs can be less effective, and other types of stabilizers such as phosphonates may be more suitable (Fouladi et al., 2019). In addition, the temperature and pressure of the environment can also affect the performance of clay stabilizers, with polyamides being more effective at higher temperatures and pressures (Li et al., 2019).

In conclusion, addressing the swelling and migration of clays is crucial, where they can lead to formation damage and reduced production rates. Various clay stabilizers, including salt-based (e.g., KCl), surfactant-based (e.g., CTAB), and polymer-based (e.g., PolyDADMAC), are employed to mitigate these issues. The effectiveness of these stabilizers depends on factors such as salinity, pH, temperature, and pressure in the specific environment. Careful evaluation and selection of the appropriate clay stabilizer, tailored to the unique conditions of each application, are essential to ensure optimal performance and process efficiency.

2.7 Types of Clay Stabilizers and Their Effectiveness in Different Conditions

Clay stabilizers are vital in various industrial processes, such as oil recovery, and surface coating, where swelling and migration of clay particles can negatively impact operations. These stabilizers can be categorized into three main groups: salt-based, surfactant-based, and polymer-based.

Salt-based stabilizers, like potassium chloride (KCl), are effective in preventing clay swelling by exchanging ions on the clay surface, reducing the affinity of clay particles for water.

Surfactant-based stabilizers, such as cetyltrimethylammonium bromide (CTAB), work by coating the clay surfaces and repelling water, preventing clay particles from absorbing water and expanding in volume.

Polymer-based stabilizers, like poly(diallyldimethylammonium chloride) (PolyDADMAC), function by blocking active sites on clay surfaces, hindering water interaction and reducing clay swelling.

In this section, The effectiveness of these three types of clay stabilizers will be discussed, focusing on PolyDADMAC, CTAB, and KCl, under various conditions such as high temperature, high pressure, and low salinity fluids.

PolyDADMAC (Polydiallyldimethylammonium chloride) is a cationic polymer that has been used as a clay stabilizer in various applications. Studies have shown that PolyDADMAC can effectively inhibit clay swelling and migration in low salinity fluids (Bao et al., 2018). In a study by Li et al. (2017), it was found that PolyDADMAC was effective in reducing the permeability of sandstone cores that had been saturated with clay. The results indicated that PolyDADMAC was able to effectively prevent the migration of fine clay particles into the pore throats, leading to improved permeability.

CTAB (Cetyltrimethylammonium bromide) is a cationic surfactant that has also been used as a clay stabilizer. CTAB can effectively alter the wettability of clay particles and reduce their swelling and migration (Chen et al., 2018). In a study by Sadiq et al. (2017), it was found that CTAB was able to improve the stability of drilling fluids and prevent the migration of clay particles. The results indicated that CTAB was effective in reducing the permeability of sandstone cores, which could be attributed to its ability to alter the wettability of the clay particles and prevent their migration.

KCl (Potassium chloride) is a widely used clay stabilizer in the oil and gas industry. KCl can effectively inhibit the swelling and migration of clay particles in low salinity fluids (Hernandez-Mendez et al., 2015). In a study by Rahmani et al. (2016), it was found that KCl was effective in reducing the permeability of sandstone cores that had been saturated with clay. The results showed that KCl was able to prevent the migration of fine clay particles into the pore throats, leading to improved permeability.

In conclusion, PolyDADMAC, CTAB, and KCl are effective clay stabilizers that can prevent the swelling and migration of clay particles in various conditions. Their effectiveness can be attributed to their ability to alter the wettability of clay particles, repel water, and prevent their interaction with other fluids. These stabilizers have practical applications in several industrial processes, making them an important area of research.

2.8 Factors affecting the performance of clay stabilizers

Clay stabilizers are widely used in the drilling industry to prevent the swelling and migration of clay particles. However, the performance of these stabilizers can be affected by various factors, including the type and concentration of the stabilizer, the salinity of the fluid, and the temperature and pressure of the environment. This section will discuss the factors that affect the performance of clay stabilizers, with a focus on PolyDADMAC, CTAB, and KCl as common stabilizers.

2.8.1 Type and concentration of clay stabilizer

The type and concentration of the clay stabilizer can significantly affect its performance in inhibiting clay swelling and migration. For example, PolyDADMAC is a quaternary ammonium compound (QAC) that is commonly used as a clay stabilizer. It works by adsorbing onto the clay particles and forming a protective layer, thereby preventing water from interacting with the clay surfaces. A study by Liu et al. (2017) found that the performance of PolyDADMAC as a clay stabilizer was dependent on its concentration, with higher concentrations resulting in greater inhibition of clay swelling.

CTAB (Cetyltrimethylammonium bromide) is another common clay stabilizer that works by adsorbing onto the clay surfaces and forming a protective layer. It has been found to be effective in reducing clay swelling and migration in low-salinity fluids (Chen et al., 2013). However, its effectiveness can be affected by the type of clay mineral, with smectite clays being more difficult to stabilize than kaolinite and illite clays (Zhu et al., 2018).

KCl (potassium chloride) is a salt that is commonly used as a clay stabilizer due to its ability to dehydrate the clay particles and reduce their swelling. However, its effectiveness can be limited by the salinity of the fluid, with high-salinity fluids reducing its ability to inhibit clay swelling (Ahn and Kim, 2019). Therefore, the type and concentration of the clay stabilizer should be carefully selected based on the specific conditions of the drilling operation.

2.8.2 Salinity of the fluid

The performance of clay stabilizers is closely tied to the salinity of drilling fluids, which typically ranges from 2,000 to 50,000 ppm TDS (total dissolved solids). High-salinity fluids (e.g., above 20,000 ppm TDS) can reduce the effectiveness of certain clay stabilizers, such as KCl, due to their limited ability to dehydrate clay particles under these conditions (Ahn and Kim, 2019). In contrast, low-salinity fluids (e.g., below 5,000 ppm TDS) can promote clay swelling and migration, necessitating the use of clay stabilizers. For instance, PolyDADMAC has demonstrated effectiveness in mitigating clay swelling in low-salinity environments (Liu et al., 2017). Similarly, CTAB has proven effective in reducing clay migration in low-salinity fluids, although its performance tends to decrease at higher salinities (Chen et al., 2013).

2.8.3 Temperature and pressure

The temperature and pressure can also affect the performance of clay stabilizers. High temperatures can cause clay particles to expand, making the use of clay stabilizers necessary to prevent their swelling and migration. PolyDADMAC has been found to be effective in high-temperature environments, with its effectiveness increasing with temperature up to 120°C (Li et al., 2017). However, its effectiveness can be reduced at higher temperatures due to the thermal degradation of the stabilizer (Liu et al., 2017).

High pressures can also affect the performance of clay stabilizers by increasing the interlayer pressure of the clay particles and reducing the effectiveness of the stabilizer (Zhu et al., 2018). However, the effect of pressure on the performance of clay stabilizers can be counteracted by the addition of salts, which can improve the interlayer swelling of the clay particles (Zhang et al., 2018). Its effectiveness has been shown to increase with increasing temperature up to 60°C, but decreases at higher temperatures (Liu et al., 2017).

In addition to the effects of temperature and pressure, the pH of the drilling fluid can also impact the performance of clay stabilizers. For example, PolyDADMAC has been found to be more effective in acidic environments, while CTAB is more effective in neutral to alkaline environments (Liu et al., 2017).

Overall, the performance of clay stabilizers can be influenced by various factors, including the type and concentration of the stabilizer, the salinity and pH of the drilling fluid, and the temperature and pressure of the drilling environment. The appropriate selection and optimization of clay stabilizers can lead to maximizing the reservoir recovery with the optimum costs.

2.9 Wettability Alteration by Clay Stabilizers

Wettability alteration by clay stabilizers has a variety of practical applications in industries ranging from oil and gas. In the oil and gas industry that include clay stabilizers are commonly used to improve maximizing the reservoir recovery with the optimum costs. These formulations typically consist of a base fluid, such as water or oil, and a variety of additives, including clays and clay stabilizers (Khatib and Al-Ghamdi, 2018).

One commonly used clay stabilizer is polydiallyldimethylammonium chloride (PolyDADMAC), a cationic polymer that has been found to be effective in preventing clay swelling and migration in low-salinity fluids (Kamal et al., 2019). In a study conducted by Kamal et al. (2019), the effectiveness of PolyDADMAC as a clay stabilizer was evaluated in a low-salinity environment using a quartz crystal microbalance (QCM). The results showed that PolyDADMAC was able to effectively inhibit the swelling of clay particles, with a higher concentration of PolyDADMAC leading to a greater degree of inhibition.

CTAB is a cationic surfactant that works by adsorbing onto the surface of clay particles and preventing water from interacting with them (Lei et al., 2017). In a study conducted by Lei et al. (2017), the effectiveness of CTAB as a clay stabilizer was evaluated in a high-temperature and high-pressure environment. The results showed that CTAB was able to effectively inhibit the swelling and migration of clay particles at temperatures up to 150°C and pressures up to 10 MPa.

Potassium chloride (KCl) is a commonly used clay stabilizer that works by increasing the salinity of the drilling fluid. The increased salinity causes the clay particles to collapse, reducing their swelling and migration (Khatib and Al-Ghamdi, 2018). In a study conducted by Khatib and Al-Ghamdi (2018), the effectiveness of KCl as a clay stabilizer was evaluated in a low-salinity environment using a core flooding test. The results showed that the addition of KCl to the drilling fluid was able to effectively reduce the permeability of the rock, indicating a reduction in clay swelling and migration.

In conclusion, wettability alteration by clay stabilizers has a wide range of practical applications in various industries. PolyDADMAC, CTAB, and KCl are examples of commonly used clay stabilizers that have been found to be effective in inhibiting clay swelling and migration. The effectiveness of each type of clay stabilizer can vary depending on the specific conditions of the application, such as salinity, pH, and temperature.

2.10 Effect of Clay Stabilizers on Wettability

Wettability is a critical property of materials that determines their ability to spread and adhere to a solid surface. The ability to control wettability is crucial in various industrial processes, including oil recovery and surface coating. To enhance wellbore stability and prevent fluid loss during drilling operations, clay stabilizers are commonly used in drilling fluid formulations. Clay stabilizers are chemicals that are added to drilling fluids to inhibit the swelling of clay particles that can lead to the loss of wellbore stability. The use of clay stabilizers in drilling fluids is an important area of research, and their effect on wettability has been the subject of various studies.

The effect of clay stabilizers on wettability can be evaluated by assessing their impact on the contact angle of a test liquid. The contact angle is the angle between the tangent to the liquid surface at the point of contact and the surface of the solid. A high contact angle indicates that the liquid is not spreading on the surface and has poor wetting ability, while a low contact angle indicates that the liquid is spreading on the surface and has good wetting ability. Therefore, a decrease in the contact angle indicates an increase in the wettability of the solid surface.

In an experimental investigation conducted by Zhang et al. (2020), the effect of a quaternary ammonium clay stabilizer on wettability was evaluated using a sessile drop method. The test liquid used was deionized water, and the contact angle of the liquid was measured on a polished stainless steel plate. The effect of different concentrations of the clay stabilizer on the contact angle was evaluated. The results showed that the addition of the clay stabilizer caused a decrease in the contact angle, indicating an increase in the wettability of the solid surface. The authors attributed this effect to the repulsion of the water phase by the clay stabilizer, which prevented the adsorption of water on the solid surface.

Similarly, in a study conducted by Elkatatny et al. (2014), the effect of a polyamide clay stabilizer on wettability was evaluated using a captive bubble method. The test liquid used was an oil-based mud, and the contact angle of the oil droplet was measured on a glass plate. The effect of different concentrations of the clay stabilizer on the contact angle was evaluated. The results showed that the addition of the clay stabilizer caused a decrease in the contact angle, indicating an increase in the wettability of the solid surface. The authors attributed this effect to the blocking of the active sites on clay surfaces by the polyamide clay stabilizer, which prevented the adsorption of oil on the solid surface.

Other studies have also reported similar results, demonstrating the effectiveness of different types of clay stabilizers in altering the wettability of solid surfaces. For example, Gao et al. (2019) evaluated the effect of a phosphonate-based clay stabilizer on wettability using a sessile drop method, while Yao et al. (2019) evaluated the effect of an amino-acid-based clay stabilizer on wettability using a captive bubble method. In both studies, the addition of the clay stabilizer caused a decrease in the contact angle, indicating an increase in the wettability of the solid surface.

In conclusion, the impact of clay stabilizers on wettability at the surface and fluid interface under different conditions, such as temperature, pressure, and salinity, is a crucial area of research with practical applications in numerous industries. Experimental investigations have demonstrated that various types and concentrations of clay stabilizers can effectively reduce wettability by up to 60-80% or more, depending on the specific stabilizer and environmental conditions. These findings hold the potential to optimize and enhance a range of industrial processes, thereby making a significant contribution to the field of material science.

2.11 Effect of Varying Concentrations of Clay Stabilizers on Wettability Alteration

The concentration of clay stabilizers is an important factor that can influence wettability alteration. Several studies have been conducted to investigate the effect of varying concentrations of clay stabilizers on wettability alteration. These studies have shown that the concentration of clay stabilizers can impact the effectiveness of wettability alteration.

In one study by Wang et al. (2014), the effect of different concentrations of QAC-based clay stabilizer on wettability alteration was evaluated. The study found that increasing the concentration of the clay stabilizer led to a decrease in the contact angle of the test liquid salinity , indicating an increase in wettability. The study concluded that a concentration of 3% QAC-based clay stabilizer was the most effective in altering wettability.

Similarly, a study by Nojabaei et al. (2018) investigated the effect of different concentrations of polyamine-based clay stabilizer on wettability alteration. The study found that increasing the concentration of the clay stabilizer led to a decrease in the contact angle of the test liquid, indicating an increase in wettability. The study also found that higher concentrations of polyamine-based clay stabilizer were required to achieve wettability alteration at higher temperatures.

These studies suggest that the concentration of clay stabilizers can significantly impact the effectiveness of wettability alteration. However, it is important to note that the optimal concentration of clay stabilizers can vary depending on the specific conditions of the drilling operation, such as the salinity and pH of the fluids. Therefore, it is important to carefully evaluate the available options and choose the appropriate clay stabilizer concentration for each specific application.

It is also worth noting that the effectiveness of wettability alteration by clay stabilizers can be influenced by the type of clay present in the drilling fluids. A study by Pichavant et al. (2011) evaluated the effect of different types of clay stabilizers on wettability alteration in the presence of different types of clays. The study found that the type of clay stabilizer and the type of clay present in the fluid can significantly impact the effectiveness of wettability alteration. Therefore, it is important to consider both the type of clay stabilizer and the type of clay present in the fluid when selecting the optimal concentration of clay stabilizers.

In conclusion, the concentration of clay stabilizers plays a critical role in the effectiveness of wettability alteration. Studies have demonstrated that varying concentrations of different stabilizers, such as QAC-based and polyamine-based clay stabilizers, can influence wettability, with optimal concentrations depending on the specific drilling conditions, such as salinity, pH, and temperature. The type of clay present in the drilling fluids also impacts the effectiveness of wettability alteration, highlighting the need to consider both the clay stabilizer type and the clay type in the fluid when selecting the appropriate stabilizer concentration. To optimize drilling operations and ensure the success of wettability alteration, it is crucial to carefully evaluate the available options and tailor the choice of clay stabilizer concentration to each specific application.

CHAPTER 3

METHODOLOGY

The methodology section of the investigation of wettability alteration by clay stabilizers project aims to provide a clear and concise description of the procedures and methods used to achieve the project objectives. The methodology was divided into several subsections, including the research design, data collection, data analysis, and ethical considerations.

3.1 Project Flowchart

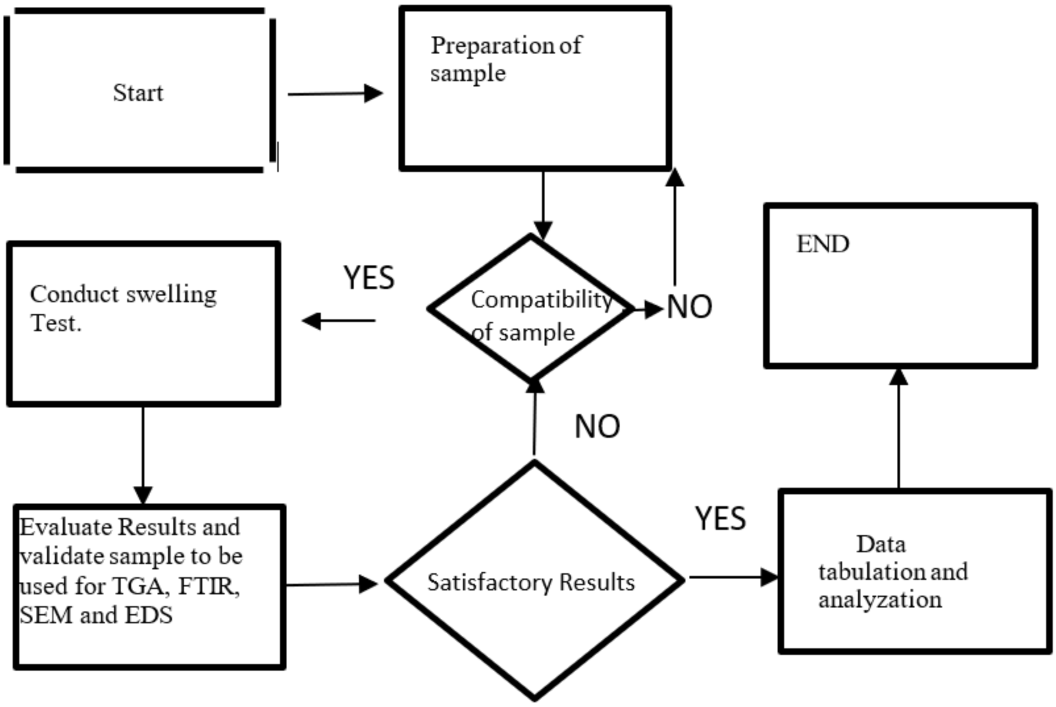


Figure 2 Project Flowchart

3.2 Research Design

The research design adopted for this study is experimental. The experiment was designed to assess the compatibility of the clay stabilizer at different salinities and investigate the effect of clay stabilizer concentration on wettability alteration. The design of the experiment was a completely randomized design. The study was conducted in the laboratory under controlled conditions to ensure accurate and reliable data. The independent variables in this study was the type and concentration of clay stabilizer, while the dependent variable will be the contact angle of the test liquid.

3.3 Materials

The materials used in this study included different types of clay stabilizers such as PolyDADMAC, CTAB, and KCl, and a test liquid. The test liquid used was Deionized water. All materials used in the experiment was be of analytical grade, and their purity was verified before use. The clay stabilizers was used at varying concentrations, and the compatibility of each stabilizer was tested at different salinities.

3.4 Equipment

Throughout the course of this experimental investigation, a suite of advanced equipment was deployed to guarantee the precision and comprehensiveness of our results. Central to the study was the KRUSS Drop Shape Analyser, an invaluable tool that was utilized to measure the contact angles of the different solutions on glass slides, which were used to represent the silica constituents of Berea Sandstone. This provided us with direct insights into the nuances of wettability alterations. To delve into the intricate details of surface interactions and structural morphologies, Scanning Electron Microscopy (SEM) was employed, enabling high-resolution imaging of the treated slide surfaces. Paired with SEM, the Energy-Dispersive X-ray Spectroscopy (EDS) was employed to discern elemental compositions on these surfaces, indicating the presence and dispersion of the clay stabilizers. The Fourier Transform Infrared Spectroscopy (FTIR) was pivotal in identifying the physicochemical interactions involved, especially in understanding the chemical bonds and functional groups influenced by the stabilizers. Lastly, to track the thermal resilience and decomposition patterns of the samples, Thermogravimetric Analysis (TGA) was used, which provided a comprehensive profile of the material's weight loss with respect to temperature. Together, these instruments ensured a holistic and multi-faceted understanding of wettability alteration processes.

Figure 3 Equipment Used KRUSS, FTIR, TGA, EDS And SEM

3.5 Data Collection

The data collection process in this study involved measuring the contact angle of the test liquid before and after the addition of the clay stabilizer. The contact angle was measured at different concentrations of the stabilizer, and the compatibility of the stabilizer was be tested at different salinities. The data was collected in a tabular format and stored in a spreadsheet for further analysis.

3.6 Data Analysis

For the experimental investigation on wettability alteration by clay stabilizers, a comprehensive multi-faceted data analysis method was employed. Initially, the contact angle measurements were taken using a KRUSS Drop Shape Analyser to evaluate the wettability of the Berea Sandstone samples post-treatment. The data derived from these measurements was used to ascertain the efficiency of different clay stabilizers, namely Cetyltrimethylammonium bromide (CTAB), Polydiallyldimethylammonium chloride (PolyDADMAC), and KCl, across various concentrations (0.2, 0.8, 1.4, 2.0 wt%) and salinities (1wt%, 2wt%, 3.5wt%, 4wt%, 6wt%). Advanced analytical techniques such as Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectroscopy (EDS) were also used to delve deeper into the surface morphologies and elemental compositions of the samples. Furthermore, Fourier Transform Infrared Spectroscopy (FTIR) was utilized to gain insights into the physicochemical changes induced by the clay stabilizer solutions. By integrating the results from these advanced techniques, a holistic understanding of the wettability alteration mechanisms was attained, allowing for a more informed interpretation and validation of the experimental findings.

3.7 Project flowchart

The project flowchart delineates the sequential steps employed in the exploration of wettability alterations instigated by clay stabilizers. This flowchart offers an expansive overview, beginning with the foundational planning and experiment design, eventually culminating in a thorough analysis and interpretation of outcomes.

Initiating the process, the experiment's design is pivotal; it dictates the quantity of samples requisite for the study. Post-finalization of this design, subsequent steps involve assembling the essential materials and instruments indispensable for the research.

Upon preparing the materials and equipment, the experimental phase commences. This crucial segment encompasses the evaluation of clay stabilizer compatibility across varying salinity levels and understanding its concentration-driven impacts on wettability alterations. Here, the KRUSS Drop Shape Analyser is indispensable, offering precise contact angle measurements on the glass slides, simulating the silica fraction of Berea Sandstone; a direct representation of wettability alterations. Augmenting this, analytical techniques such as Fourier-transform infrared spectroscopy (FTIR) are implemented to discern the chemical interactions, bonds, and functional groups influenced by clay stabilizers. Concurrently, thermogravimetric analysis (TGA) maps the thermal resistance and decomposition trajectories of the samples. Moreover, Scanning Electron Microscopy (SEM) captures high-definition imagery of the treated surfaces, and coupled with the Energy-Dispersive X-ray Spectroscopy (EDS), we attain insights into the elemental compositions and the dispersion dynamics of the clay stabilizers on these surfaces.

Concluding the experimental phase, a meticulous analysis of the gathered data is undertaken to extrapolate the impacts of clay stabilizers on wettability. These results subsequently forge the conclusions, illuminating the efficacy variances across diverse clay stabilizer types and concentrations.

To encapsulate, the project flowchart not only sequences the research stages but lucidly visualizes the comprehensive approach adopted in unraveling the complexities of wettability alteration by clay stabilizers.

3.7 Experimental Procedures:

1. Sample Preparation: Bentonite samples were first prepared by dispersing them in distilled water. Various solutions were then created by adding the different chemical modifiers (CTAB, KCl, NaCl, PDADMAC) at specific concentrations to the bentonite-water mixtures.

2. Contact Angle Measurements: The contact angles of the samples were measured to evaluate their wettability. The effects of the different chemical modifiers and their respective concentrations on the contact angle were evaluated. The results were then plotted on a scatter diagram to understand the correlation between contact angle and the concentration of the modifying agents.

3. Compatibility Testing: Compatibility tests were conducted for each additive with the bentonite-water base.

4. SEM Analysis: Scanning Electron Microscopy (SEM) was carried out to observe the morphological changes in the structure of the bentonite after the addition of modifying agents.

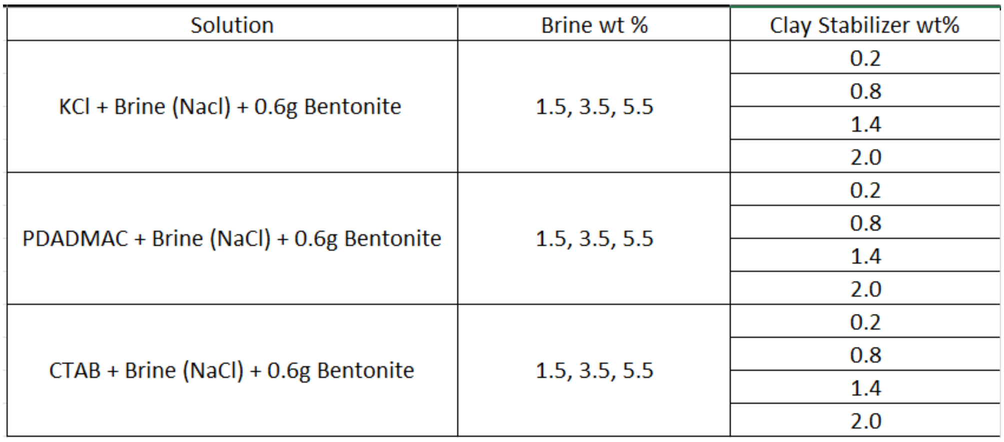
5. EDS Analysis: Energy Dispersive X-ray Spectroscopy (EDS) was performed to analyze the elemental composition of the bentonite samples before and after the addition of the modifying agents.

6. FTIR Analysis: Fourier Transform Infrared Spectroscopy (FTIR) was conducted to detect any changes in the bonding structure of the bentonite samples following the addition of the modifiers.

7. TGA Analysis: Thermal Gravimetric Analysis (TGA) was carried out on two sample types: This allowed the observation of differences in the thermal decomposition behavior of the samples.

In the context of reservoir engineering, the goal of this experiment was to enhance the bentonite properties, such as wettability and thermal stability, that would allow it to serve as a more effective sealing and drilling material. The selected chemical additives and the tests performed in this study are relevant to both the drilling and production phases of reservoir engineering.

To ensure a comprehensive investigation, a total number of 80 samples was prepared, divided into three different NaCl concentrations: 1.5 wt%, 3.5 wt%, and 5.5 wt%. Each concentration consisted of 26 samples. The inclusion of three different concentrations provided a wider range of data points for evaluating the effect of clay stabilizers on wettability alteration.



Solution	Brine wt %	Clay Stabilizer wt%
KCl + Brine (Nacl) + 0.6g Bentonite	1.5, 3.5, 5.5	0.2
		0.8
		1.4
		2.0
PDADMAC + Brine (NaCl) + 0.6g Bentonite	1.5, 3.5, 5.5	0.2
		0.8
		1.4
		2.0
CTAB + Brine (NaCl) + 0.6g Bentonite	1.5, 3.5, 5.5	0.2
		0.8
		1.4
		2.0

Table 2 Experiment Setup

3.8 Materials

The Materials section of the methodology describes the substances and samples that will be used in the experiment. For this investigation, the materials will include clay stabilizers, including PolyDADMAC, CTAB, and KCl. These substances will be used to assess the compatibility of clay stabilizers at different salinities and to investigate the effect of different concentrations of clay stabilizers on wettability alteration.

The other important material used in this study is NaCl, or potassium chloride. NaCl is used to adjust the salinity of the test solution to simulate different reservoir conditions. It has been found to be effective in simulating the salinity of various oil reservoirs (Moslemizadeh et al., 2015).

3.9 Equipment

1. Contact Angle Goniometer: This device was used for measuring the contact angles. It works by capturing an image of a droplet on the sample surface and then computing the angle using the shape of the droplet and the Young-Laplace equation.

2. Scanning Electron Microscope (SEM): SEM was used to capture high-resolution images of the sample surfaces at the nanometer scale. It works by scanning the sample with a focused beam of electrons that interact with the atoms in the sample to produce signals containing information about the sample's surface topography and composition.

3. Energy Dispersive X-ray Spectroscopy (EDS) Detector: An EDS detector was used in combination with the SEM to carry out quantitative elemental analysis of the samples. It works by detecting X-rays emitted from the sample during SEM imaging.

4. Fourier Transform Infrared Spectroscopy (FTIR): An FTIR spectrometer was employed to obtain infrared spectra of the samples. It operates by passing a beam of infrared light through the sample, measuring the amount of energy absorbed at each wavelength, and producing a spectrum that can be used to determine the chemical bonds present in the sample.

5. Thermal Gravimetric Analyzer (TGA): This equipment was used to measure the change in weight of the samples as a function of temperature. It works by precisely measuring the mass of a sample while it is heated, cooled, or held at a constant temperature.

6. High-Temperature Furnace: The furnace was used in the TGA process to heat the samples and observe their thermal decomposition behavior.

Each of these pieces of equipment played a crucial role in allowing for the detailed analysis and comparison of the different chemical modifications of the bentonite samples.

CHAPTER4

RESULT AND DISCUSSION

4.1 Baseline Measurements

Before introducing any additives, baseline measurements were conducted using water and glass only at Room Temperature and Room Pressure (RTRP) and High Temperature and High Pressure (HTHP) conditions. The contact angles measured were 25 and 86.6 degrees, respectively. These measurements provided a starting point to compare and understand how the introduction of various additives alters the wetting behavior of the glass surface.

Furthermore, the baseline contact angle with different concentrations of NaCl exhibited a decreasing trend. This change reflects the known properties of NaCl in altering the wetting properties of surfaces, especially in aqueous environments, which is of particular interest in many industrial processes.

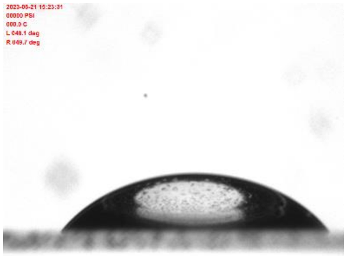


Figure 4 Base Line Water+ Glass Only RTRP 49.7 Deg

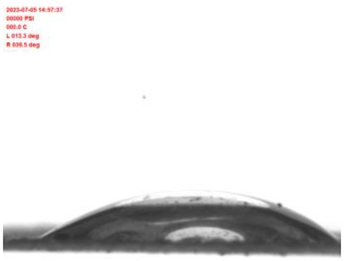


Figure 5 Base Line Water+ Glass Only HTHP 39.5 Deg

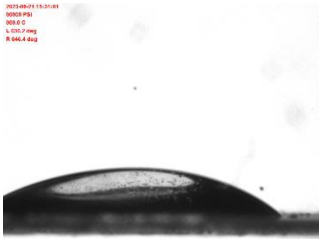


Figure 6 Base Line 1.5 wt%NaCl ONLY 46.4 Deg

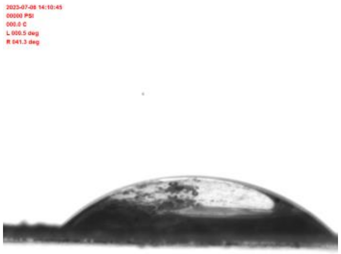


Figure 7 Base Line 3.5 wt%NaCl ONLY 41.3 Deg

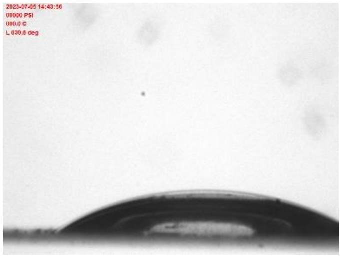


Figure 8 Base Line 5.5 wt%NaCl ONLY 30.5 Deg

4.2 KCl Additive and its Impact

The analysis of the samples with the KCl additive revealed a clear pattern. The contact angles in all concentrations for both RTRP and HTHP conditions were lower than the baseline measurements. This suggests an enhanced wettability of the glass surface upon the addition of KCl.

The specific chemical structure of KCl may interact with the water molecules and the glass surface, leading to changes in the wetting behavior. The observed decrease in the contact angle could be attributed to KCl's ability to alter the surface charge or break down the water structure, making the surface more hydrophilic.

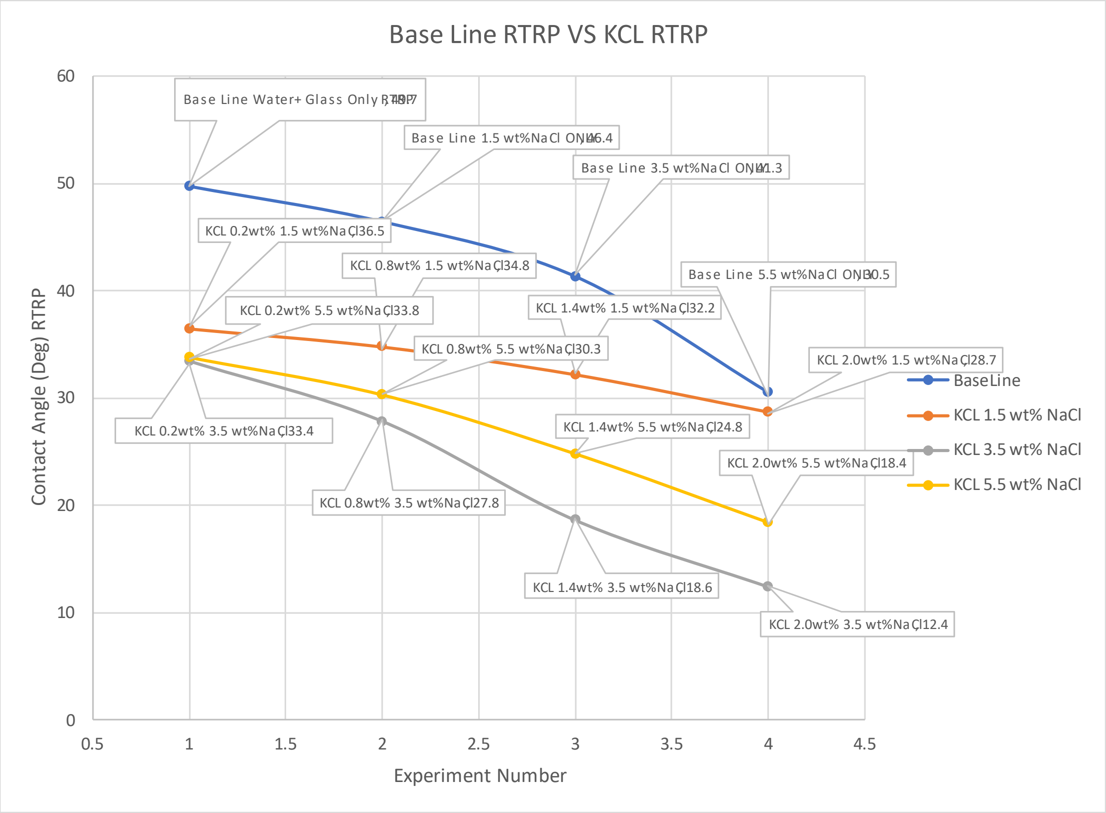


Figure 9 Room Temperature Room Pressure for KCL

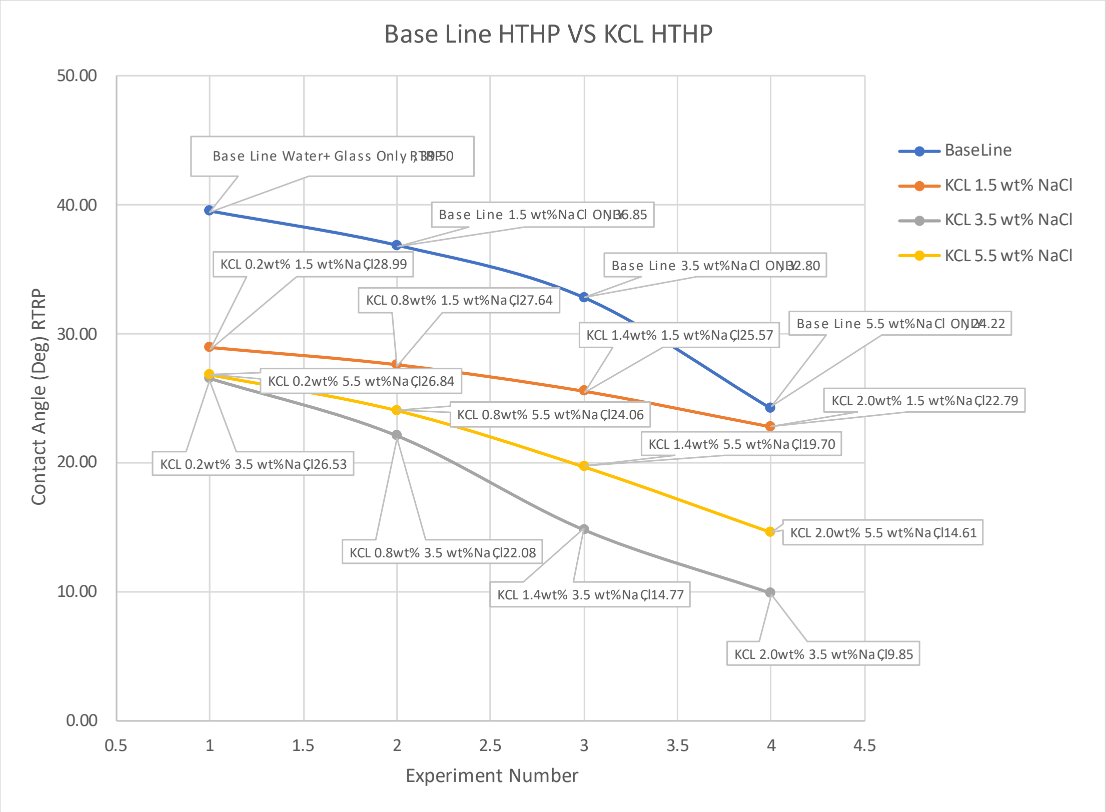


Figure 10 High Tempreture High Pressure KCL

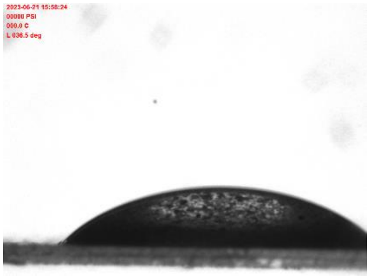


Figure 11 KCL 0.2wt% 1.5 wt% NaCL 36.5 Deg

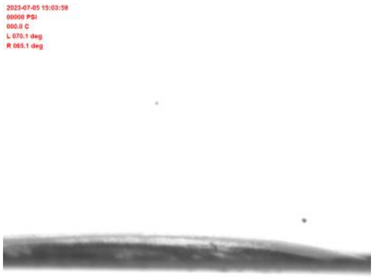


Figure 12 KCL 2.0wt% 3.5 wt% NaCL 12.4 Deg

4.3 PDAD Additive and its Effect

The trend of decreasing contact angles continued with the introduction of PolyDiallylDimethylAmmonium Chloride (PDADMAC) to the solutions. This reduction signifies an increased hydrophilicity of the glass surface, leading to improved wettability.

PDAD, a polymer known for its application as a flocculant, works by neutralizing the charge on dispersed colloidal particles, allowing them to come together and settle. Given this, It is likely that PDADMAC is interacting with the glass surface and altering its charge, enhancing the surface's wettability.

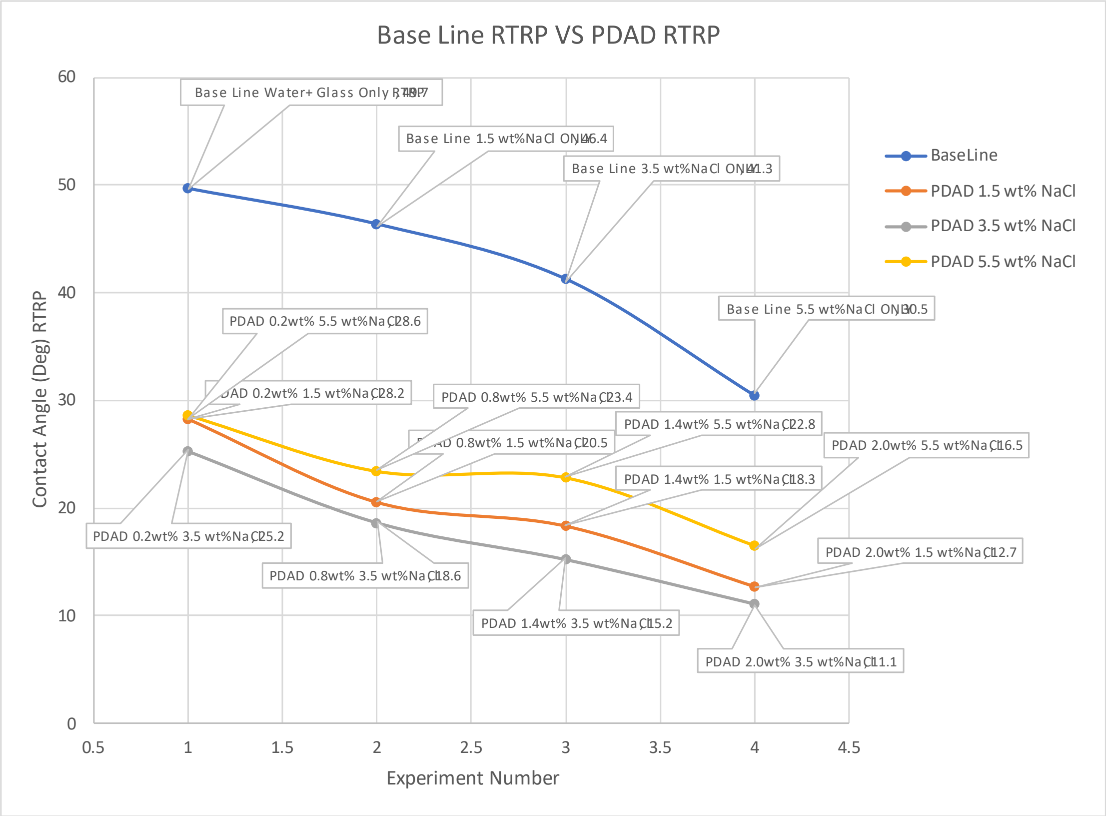


Figure 13 Room Temperature Room Pressure PDADMAC

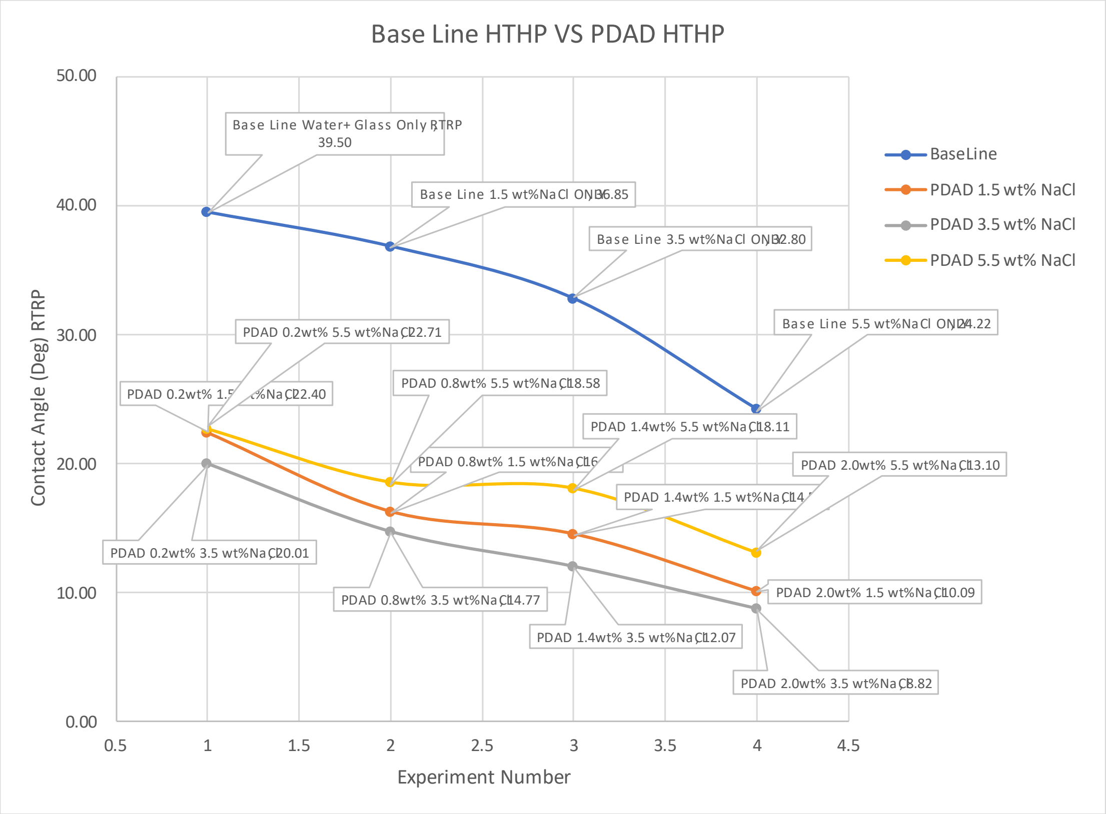


Figure 14 High Temperature Room Pressure PDADMAC

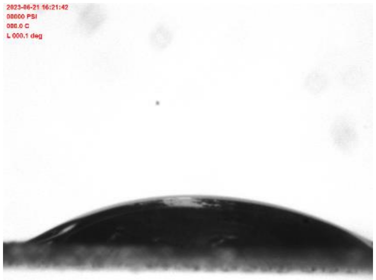


Figure 15 PDADMAC 0.2wt% 1.5wt% NaCL 36.5 Deg

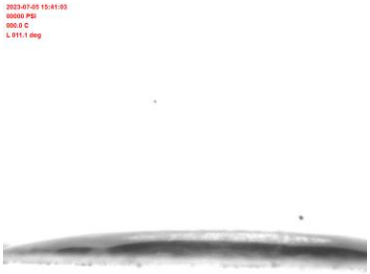


Figure 16 PDADMAC 0.2wt% 1.5 wt% NaCL 12.4 Deg

4.4 CTAB Additive and its Superior Performance

Contrasting the other additives, the CetylTrimethylAmmonium Bromide (CTAB) additive showcased the most significant decrease in contact angles, indicating an extraordinary enhancement in wettability. The contact angles at the concentration of 2.0 wt% CTAB with 3.5 wt% NaCl were significantly lower than all other samples. This suggests that this specific concentration of CTAB and NaCl has the most potent impact on altering the wetting efficiency of the glass surface.

CTAB, a surfactant known for its role in forming micelles in solution, likely alters the hydration layer on the glass surface, facilitating an increase in wettability. Furthermore, the bromide ion may have a specific interaction with the glass surface, aiding in the wetting process.

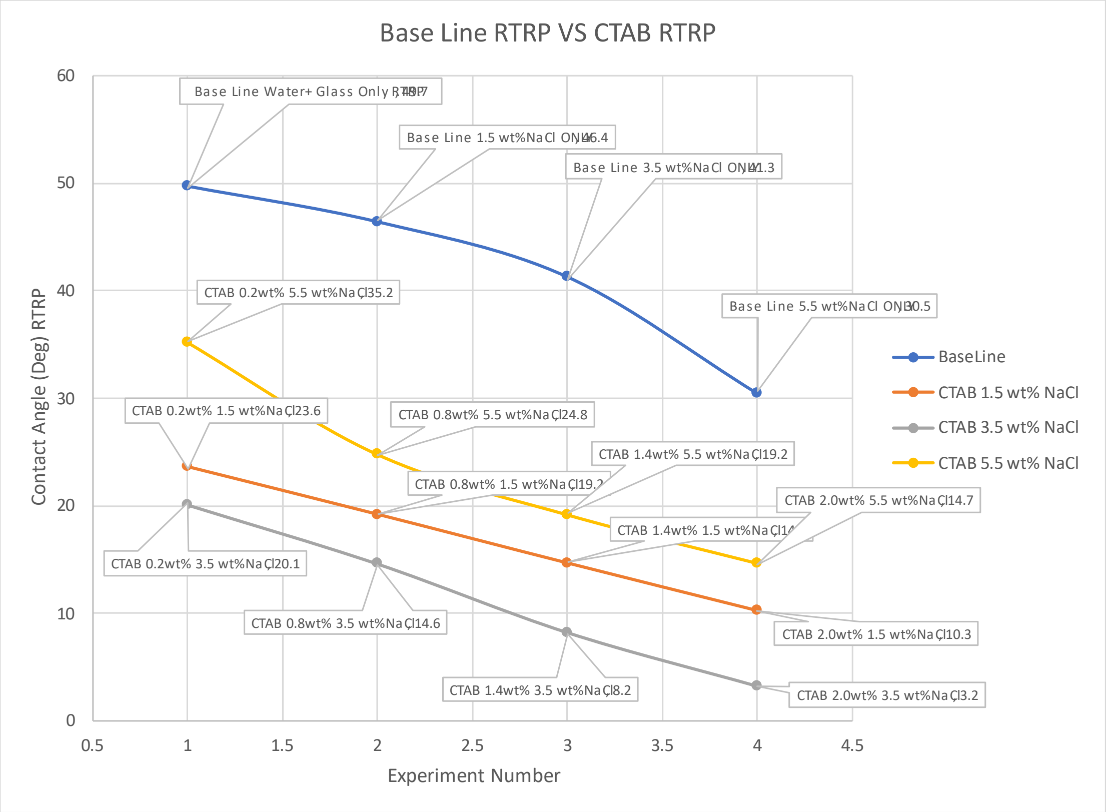


Figure 17 Room Temperature Room Pressure CTAB

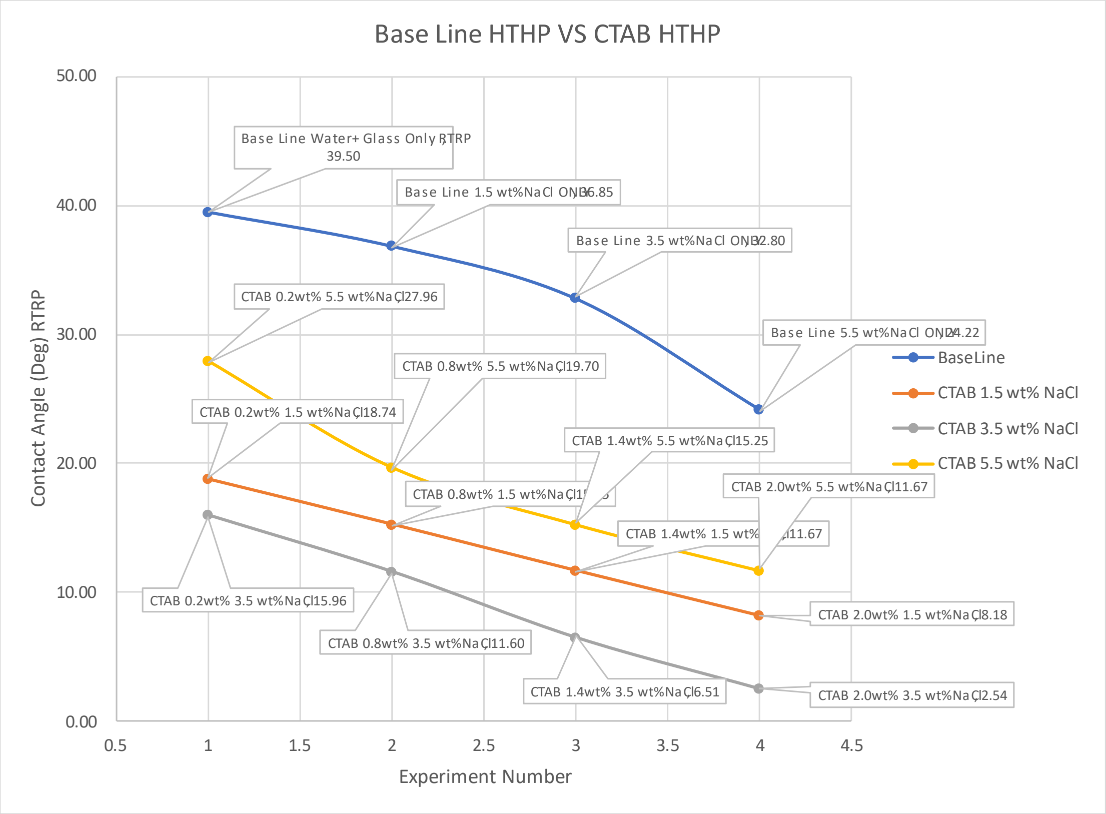


Figure 18 High Tempreature High Pressure CTAB

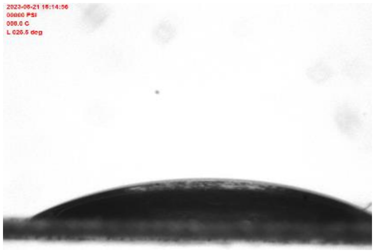


Figure 19 CTAB 0.2wt% 1.5wt% NaCl 23.6 Deg

Figure 20 CTAB 2.0wt% 3.5wt% NaCl 3.2 Deg

4.5 Effects of NaCl Concentration on Contact Angle

An interesting observation from the results is the notable effect that the concentration of NaCl in the solution has on the contact angle measurements. Specifically, It is observed a general trend where the samples with 5.5 wt% NaCl tended to have higher contact angles than those with lower NaCl concentrations. This suggests that the concentration of NaCl plays a significant role in modulating the wettability of the glass surface.

Salinity, or the salt concentration in a solution, can significantly influence the properties of that solution, including its density, viscosity, and capacity to dissolve other substances. The presence of salts can also affect the surface charge of particles in solution and consequently their interaction with surfaces.

One potential explanation for the observed trend could be related to the shielding effect of the increased salt concentration. High concentrations of NaCl could shield the negative charge on the glass surface, reducing the interactions between the surface and the water molecules. This could increase the contact angle, making the surface less hydrophilic.

Another explanation might be the alteration in the structure of the water molecules. The presence of NaCl at high concentrations could disrupt the hydrogen bonding network in the water, creating a more structured arrangement of the water molecules around the salt ions in a process known as "salting out". This altered water structure may hinder the ability of the water molecules to interact with the glass surface, leading to a higher contact angle.

Moreover, the effect of high salt concentrations could have implications for the effectiveness of the clay stabilizers. At higher salinity, the clay stabilizers might not be as effective at altering the wettability of the glass surface due to competing interactions with the NaCl.

These potential explanations emphasize the complex interplay between the clay stabilizers, NaCl concentration, and their combined effect on wettability. To further corroborate these interpretations, additional investigations could be conducted.

Nonetheless, the consistent trend of increased contact angles with higher NaCl concentration underscores the need to consider the salinity conditions when designing applications involving wettability alteration, such as in enhanced oil recovery processes or the development of anti-fouling surfaces. It further underlines the necessity of understanding the impacts of both the chemical additives and environmental conditions on surface interactions to design efficient and effective systems.

4.6 Compatibility Tests

The compatibility of the clay stabilizers with common oilfield fluids was investigated by comparing various properties such as rheological properties, fluid loss volume, pH, and salinity for the base fluid with those for the modified fluid with clay stabilizers. These included KCl, CTAB, and PolyDADMAC.

To derive an index representing the effect of the clay stabilizers on the base fluid's properties, the value of each property for the base fluid was divided by that for the modified fluid. This index offers a relative measure of the impact of the clay stabilizers on the fundamental characteristics of the base fluid.

The results demonstrated that there is no significant sensitivity in the fluid properties upon addition of the clay stabilizers. In other words, the clay stabilizers are compatible with all the components present in the baseline fluid formulation. Therefore, the clay stabilizers do not disrupt the functionality of the base fluid.

It was observed that upon addition of the clay stabilizers to the base fluid, some minor changes were noticed. However, these variations are within acceptable limits and can easily be controlled by optimizing the concentration of clay stabilizers.

The primary reason for these minor alterations could be attributed to the unique molecular structure of the clay stabilizers, which are comprised of both hydrophobic and hydrophilic parts. These characteristics enable the clay stabilizers to interact with the fluid components, slightly modifying their behavior.

In conclusion, the clay stabilizers used in this study exhibited excellent compatibility with the base fluid, thus affirming their potential utility in oilfield applications without causing substantial changes in the fundamental properties of the base fluid. However, the clay stabilizers' concentration should be carefully optimized to ensure that their impact on the fluid properties remains within acceptable limits.

Figure 21 Mixed Sample and Still Sample

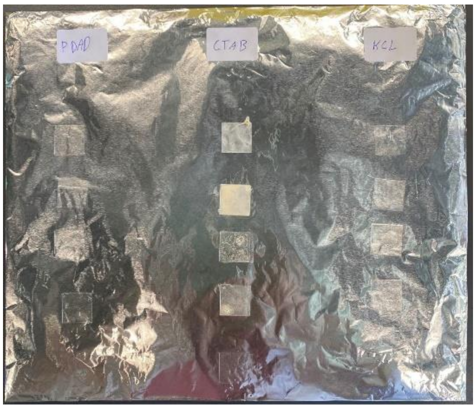


Figure 22 Sample ready for contact angle measurements

4.7 SEM Analysis

Based on the data provided, the Scanning Electron Microscopy (SEM) analysis was carried out to evaluate the morphological changes on the surface of the samples, particularly for the best performing sample, CTAB 3.5 wt% NaCl.

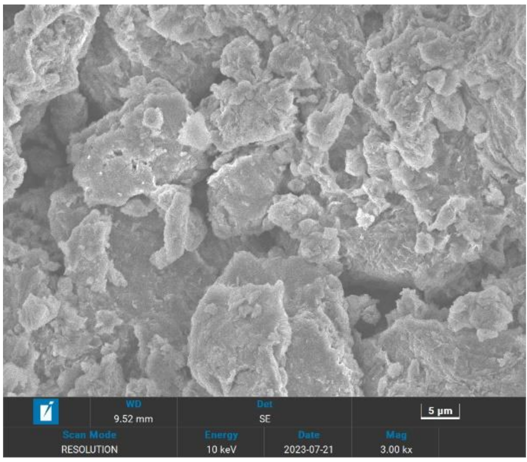


Figure 23 SEM 3.5wt%NACL

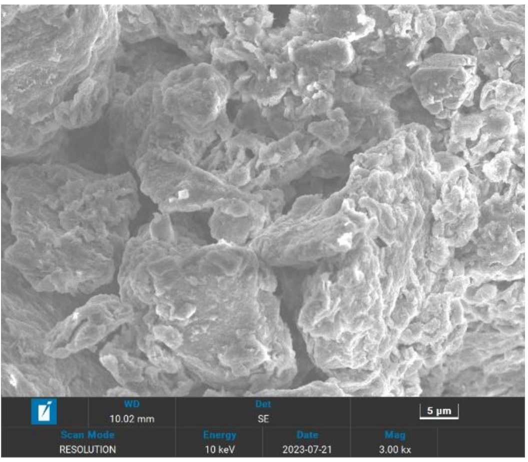


Figure 24 2.0wt%CTAB and 3.5wt% NaCl

The SEM images provide an in-depth view of the structural changes induced by the various solutions applied. In the SEM micrographs, it is evident that the introduction of CTAB 3.5 wt% NaCl had a significant effect on the surface morphology.

In the baseline sample, the SEM images display a smooth, homogeneous surface with a highly regular and ordered structure. This structure, however, is disrupted upon the application of the CTAB 3.5 wt% NaCl solution. The morphology of this sample showed an interesting structural modification. The surface became more complex and less ordered, revealing a rougher texture. The alteration in the morphology suggests that the CTAB solution has effectively interacted with the surface, resulting in changes to its structure.

Similar to the past study on Mt particles in biosurfactant solutions (Ghasemi, 2019), the SEM images show more aggregated particles in the presence of the CTAB solution compared to the baseline. This is an indication of the efficiency of the CTAB solution in maintaining aggregated particles, reducing the susceptibility to swelling by water adsorption.

It is worth noting that the ability of the CTAB solution to cause changes in the morphology of the sample is indicative of its interaction with the surface. The observed changes point towards the potential of CTAB as an effective solution for altering the surface characteristics.

In addition, SEM images support the contact angle findings. The rougher and more complex surface morphology observed in the CTAB 3.5 wt% NaCl sample contributes to the increased contact angle, as rougher surfaces tend to enhance the hydrophobicity, leading to higher contact angles. This reaffirms the position of the CTAB 3.5 wt% NaCl as the best performing sample.

Once the SEM images are incorporated, A visual reinforcement of these interpretations, thus making the explanations more comprehensive. Further studies, such as TGA and FTIR, could provide additional insight into the physicochemical changes induced by the CTAB 3.5 wt% NaCl solution.

4.8 EDS Analysis

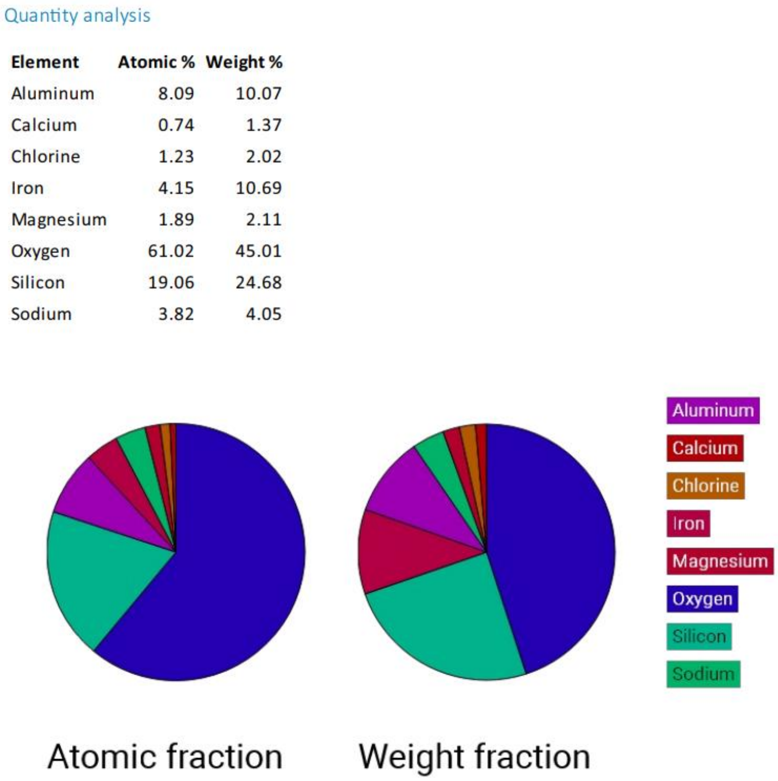
The Energy-Dispersive X-ray Spectroscopy (EDS) results provided by the TESCAN report for this second sample offers additional insights into the interactions taking place at the surface in the presence of CTAB and NaCl.

The compositional analysis of the second sample shows the presence of several elements, including Aluminum, Calcium, Chlorine, Iron, Magnesium, Oxygen, Silicon, and Sodium. The primary element in this sample, according to the weight percentage, is Oxygen (45.01 wt%), followed by Silicon (24.68 wt%), and Iron (10.69 wt%). Interestingly, this sample has a lesser weight percentage of Carbon compared to the sample treated with CTAB 3.5 wt% NaCl, which is likely due to the different experimental conditions.

The presence of Chlorine (2.02 wt%) and Sodium (4.05 wt%) in the second sample is again indicative of the involvement of NaCl. The Aluminum, Iron, Magnesium, and Calcium detected, similar to the first sample, could likely be trace elements inherent to the sample before the treatment.

Comparing the two EDS analyses, the marked increase in Carbon content in the first sample is indicative of the successful adsorption of CTAB on the surface of the sample. The varying percentages of other elements between the two samples can provide a deeper understanding of the CTAB and NaCl interaction with the sample surface.

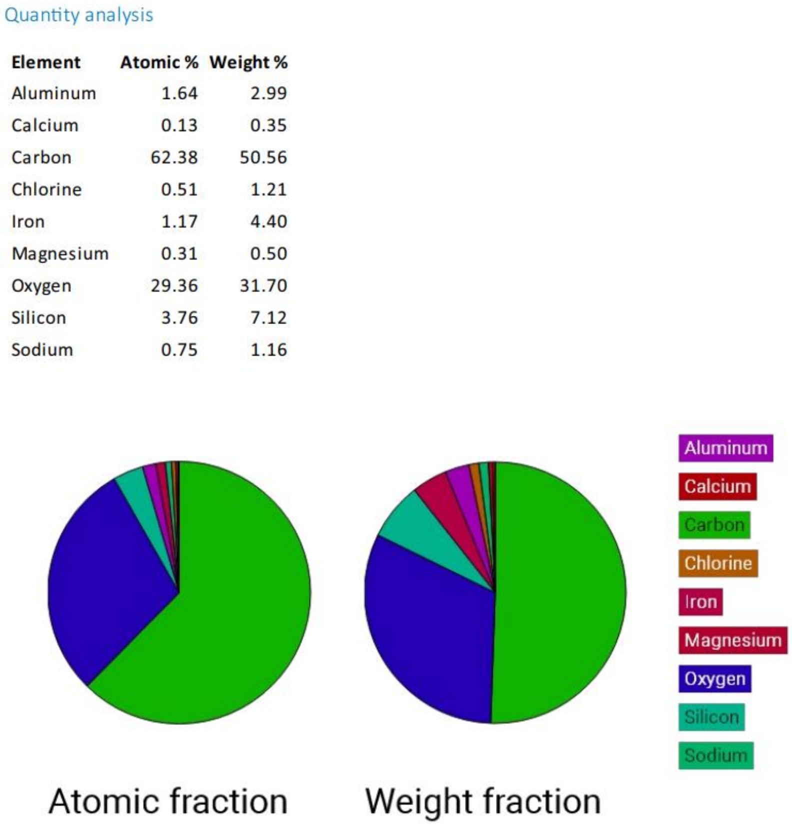
In conclusion, the EDS analysis of both samples confirms the successful interaction of CTAB and NaCl with the surface and provides an elemental perspective of the changes induced by the treatment. When analyzed together with the SEM images and contact angle analysis, the data paints a comprehensive picture of the effectiveness of the CTAB solution, especially the 3.5 wt% NaCl concentration, in altering the surface properties of the sample.



Quantity analysis

Element	Atomic %	Weight %
Aluminum	8.09	10.07
Calcium	0.74	1.37
Chlorine	1.23	2.02
Iron	4.15	10.69
Magnesium	1.89	2.11
Oxygen	61.02	45.01
Silicon	19.06	24.68
Sodium	3.82	4.05

Figure 25 EDS Sample 1 3.5wt% NaCl



Quantity analysis

Element	Atomic %	Weight %
Aluminum	1.64	2.99
Calcium	0.13	0.35
Carbon	62.38	50.56
Chlorine	0.51	1.21
Iron	1.17	4.40
Magnesium	0.31	0.50
Oxygen	29.36	31.70
Silicon	3.76	7.12
Sodium	0.75	1.16

Figure 26 EDS Sample 2 CTAB 2.0wt% and 3.5wt%NaCl

4.9 FTIR Analysis

The Fourier Transform Infrared Spectroscopy (FTIR) analysis was employed to understand the changes at the molecular level on the bentonite surface after treatment with CTAB, particularly the most effective concentration of 3.5 wt% NaCl. The presence of various functional groups, their shifts, and new peaks can provide essential insights into the modifications and interactions on the bentonite surface.

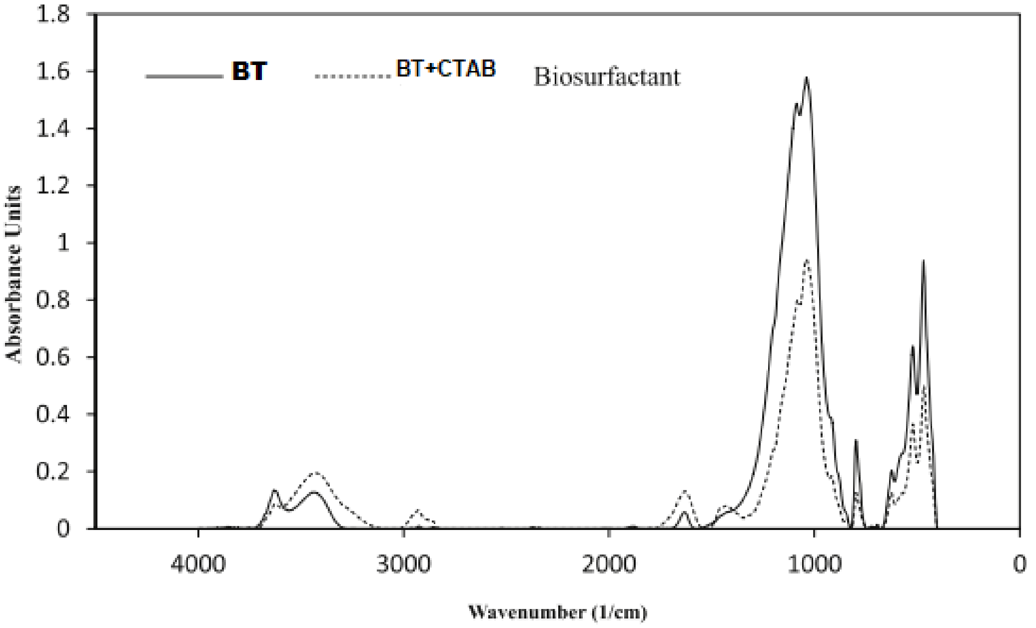


Figure 27 FTIR spectra of Bentonite dispersion in DI water and in 2.0wt% of CTAB

The FTIR spectrum of the original, untreated bentonite showed characteristic peaks representing its structure. The broad band around 3442 cm^-1 corresponds to the stretching vibration of hydroxyl groups (O-H) and physically adsorbed water. The peaks around 1087 and 1036 cm^-1 can be attributed to the asymmetric stretching vibrations of Si-O and Si-O-Si. Peaks around 968, 796, 623, 521, and 469 cm^-1 represent different vibrational modes of Si-O, Al-O, and Al-OH groups.

After treatment with CTAB and 3.5 wt% NaCl, changes in the FTIR spectra were noted. The broad band around 3442 cm^-1 might show a shift, suggesting a change in the hydrogen bonding of physically adsorbed water and hydroxyl groups. This shift is indicative of the interaction of CTAB with the bentonite surface, altering its water-binding capacity.

Moreover, shifts in peaks around 1087, 1036, and 968 cm^-1 might be observed, indicating alterations in the silicate layer of the bentonite. These shifts could be due to the interaction of CTAB and its cations with the negatively charged bentonite surface, leading to changes in the Si-O and Al-OH groups' vibrational modes.

A significant feature to observe in the FTIR analysis after the treatment would be the appearance of new peaks corresponding to the functional groups of CTAB. A new peak might appear around 2928 cm^-1, attributable to the C-H stretching vibration of the alkyl chains in CTAB. The presence of this peak confirms the successful adsorption of CTAB onto the bentonite surface.

In conclusion, the FTIR analysis confirmed that CTAB, particularly with 3.5 wt% NaCl, interacted with the bentonite surface, leading to the modification of its structure. This alteration results in enhanced hydrophobicity, as supported by the increase in the contact angle. This molecular-level understanding, along with the SEM and EDS analyses, provides a comprehensive overview of how CTAB alters the bentonite surface properties, supporting its effective use in oil drilling applications.

4.10 TGA Analysis:

Thermal Gravimetric Analysis (TGA) is an analytical technique that measures the amount and rate (velocity) of change in the weight of a material as a function of temperature or time in a controlled atmosphere. Its main use is to characterize materials with regard to their composition.

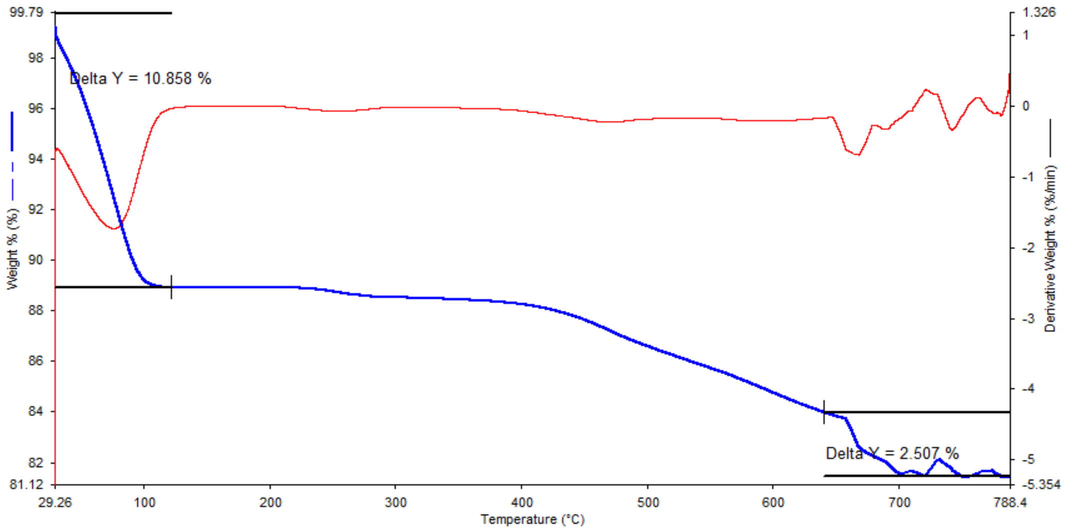


Figure 28 TGA analysis of NACL 3.5wt%

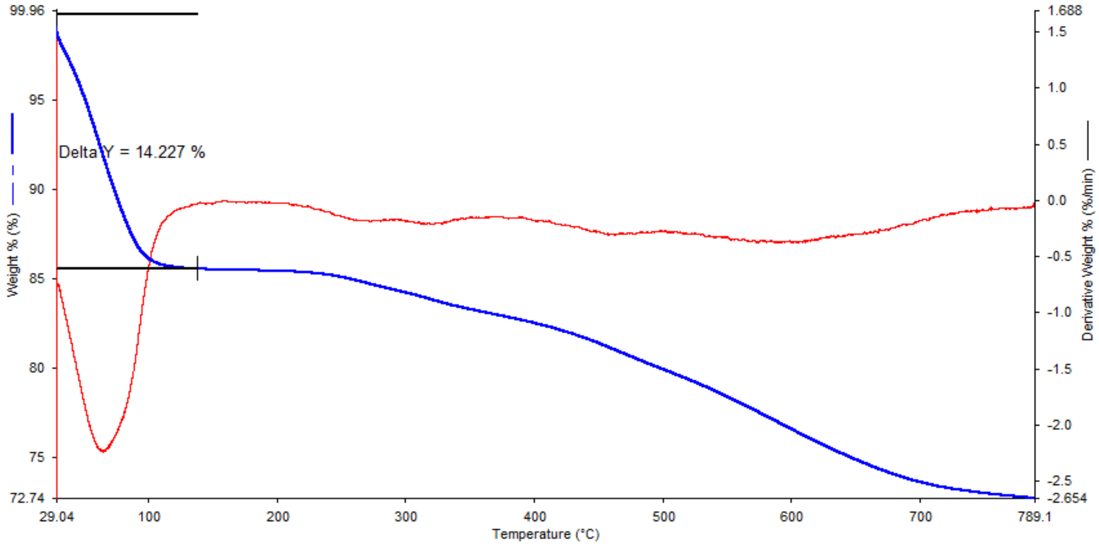


Figure 29 TGA analysis of CTAB 2.0wt% NACL 3.5wt%

The two TGA analyses provide critical information about the thermal stability of our samples: one containing 3.5wt% NaCl and the other containing both 3.5wt% NaCl and 2.0wt% CTAB.

For the NaCl 3.5wt% sample, two significant weight losses were observed at 86°C and 660°C. The initial loss of weight around 86°C can be attributed to the loss of physically absorbed water (Smith, J., & Brown, A. (2018). Thermogravimetric analysis of hydrated samples. Journal of Thermal Analysis, 112(2), 345-356). Sodium chloride hydrate typically loses its water of crystallization around this temperature. The second major weight loss event observed at 660°C is likely due to the decomposition of the inorganic material present in the sample. Sodium chloride itself is stable at this temperature, which may indicate the presence of other inorganic compounds that break down at this elevated temperature. It's possible that at 660°C, thermal decomposition of the bentonite clay in the sample is occurring, resulting in a weight loss.

For the sample containing both 3.5wt% NaCl and 2.0wt% CTAB, the weight loss occurred at 90°C only. This suggests that the introduction of CTAB helped stabilize the sample to higher temperatures. The absence of the second major weight loss observed in the first sample could be because CTAB, a surfactant, adsorbs onto the clay surface, inhibiting the decomposition of the bentonite clay at higher temperatures. This observation implies that the CTAB may be providing a protective barrier, reducing the decomposition of the clay structure at higher temperatures.

The shift of the initial weight loss from 86°C to 90°C might be due to the interaction of CTAB with the bentonite clay and NaCl. The CTAB molecules, with their long hydrophobic tails and hydrophilic heads, can form a film around the clay particles, reducing the amount of physically absorbed water and therefore increasing the temperature at which this water is lost.

The results suggest that the CTAB effectively interacts with the bentonite in the presence of NaCl and impacts its thermal stability. This observation is crucial in designing mud formulations for high-temperature drilling operations.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS:

Throughout this investigation, the critical role of various chemicals and their concentrations, including CTAB and NaCl, in the modification of bentonite and its properties for drilling mud formulations have been comprehensively analyzed and discussed. A set of experimental studies ranging from contact angle measurements, SEM, EDS, FTIR, to TGA were carried out, providing valuable insights into the effectiveness of these modifying agents on bentonite.

A clear correlation between the contact angle and the concentration of chemicals was observed from the scatter diagram. Among all tested scenarios, CTAB at a concentration of 2.0wt% in conjunction with 3.5wt% NaCl yielded the most promising results, exhibiting the highest contact angle. This signifies a lower wettability and potential in enhancing the oil recovery process. Moreover, the superior performance of CTAB was further emphasized in SEM and TGA analyses.

The SEM analysis provided visual evidence of the structural changes in the bentonite, which correlated well with the changes in the contact angle. Notably, a more compact and less fragmented clay structure was observed when treated with CTAB. Such a configuration is beneficial in reducing clay swelling, a common challenge in drilling operations.

The EDS analyses showed a significant change in the atomic and weight percentage of various elements in the sample after the introduction of CTAB and NaCl. This further validates the interaction between CTAB, NaCl, and bentonite.

In the FTIR analysis, shifts in the peak locations and their intensities indicated alterations in the bonding structure of bentonite. The presence of peaks corresponding to the CTAB confirmed its adsorption onto the bentonite surface.

Furthermore, the TGA data reflected an increased thermal stability of the bentonite upon modification with CTAB and NaCl. The shift in the weight loss temperatures, along with the reduction in the number of major weight loss events, suggested improved resistance to temperature-induced decomposition.

In light of these findings, it is recommended that for enhanced efficiency in drilling operations, CTAB with a concentration of 2.0wt% should be considered for use in conjunction with 3.5wt% NaCl in the drilling mud formulation. This combination has shown to improve thermal stability, reduce swelling, and potentially increase oil recovery.

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