Journal of Petroleum Research and Studies

journal homepage: https://jprs.gov.iq/index.php/jprs/

Print ISSN 2220-5381, Online ISSN 2710-1096

Review and Investigating Different Inhibitors for Mitigating Chemical Interaction of Shale with Drilling Mud

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Article Info

Received 22/07/2024
Revised 06/10/2024
Accepted 10/10/2024
Published 19/03/2026

DOI: http://doi.org/10.52716/jprs.v16i1.995

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Abstract

Carrying out the well drilling operations without problems is a rare process, especially those that come from drilling the shale rocks, as due to their chemical interaction with the water-based drilling mud. The objectives of this study is to present a review of most types of shale and defines the most important treatments using the shale inhibitors that are added to the drilling fluid to reduce the problem of wellbore instability. To perform a comparison with the literature, two inhibitors (potassium sorbate and potassium chloride) were used as shale inhibitors at a concentration of 4%. The efficiency of these materials was tested by using LST which shows the swelling that occurs to the shale after its interaction with the drilling mud. XRD and XRF tests also have been done to know the types and chemical compositions of the shale samples. A group of studies that proved the effectiveness of inhibitors against shale rocks were also reviewed, combining with the type of shale rocks used in each study. The results demonstrated the necessity of adding inhibition materials to reduce the shale swelling, thus reduction in non-productive time during drilling operations. Potassium sorbate inhibits shale swelling better than KCl. The swelling reached 3.938% when using 4% of potassium sorbate. As for KCl, the shale swelling reached 11.519% at same concentration. Potassium sorbate was chosen because it is an environmentally friendly and biodegradable material and through this study it was tested for the first time in Iraq as a shale inhibitor.

Keywords: Shale problems, Wellbore Instability, Drilling Problems, KCl, Potassium Sorbate.

مراجعة وفحص مثبطات مختلفة للتخفيف من التفاعل الكيميائي بين صخور السجيل وطين الحفر

الخلاصة

إن إجراء عمليات حفر الآبار دون مشاكل هي عملية نادرة، وخاصة تلك التي تأتي من حفر صخور السجيل، وذلك بسبب تفاعلها الكيميائي مع طين الحفر ذو الاساس المائي. تهدف هذه الدراسة إلى تقديم مراجعة لمعظم أنواع صخور السجيل وتحديد أهم المعالجات باستخدام مثبطات هذه الصخور التي تضاف إلى سائل الحفر لتقليل مشكلة عدم استقرار البئر. لإجراء مقارنة مع الأدبيات، تم استخدام مثبطين (سوربات البوتاسيوم وكلوريد البوتاسيوم) كمثبطات للصخور الزيتية بتركيز 4٪. تم اختبار كفاءة هذه المواد باستخدام اختبار التورم الخطي الذي

يوضح الانتفاخ الذي يحدث للصخور الزيتية بعد تفاعلها مع طين الحفر .كما تم إجراء اختبارات حيود الأشعة السينية و الأشعة السينية الفلورية لمعرفة أنواع وتركيبات عينات الصخور الزيتية الكيميائية. كما تم استعراض مجموعة من الدراسات التي أثبتت فعالية المثبطات ضد صخور السجيل. وأظهرت النتائج ضرورة إضافة مواد مثبطة لتقليل انتفاخ هذه الصخور وبالتالي تقليل الوقت غير المنتج أثناء عمليات الحفر. سوربات البوتاسيوم يثبط انتفاخ صخور السجيل بشكل أفضل من كلوريد البوتاسيوم حيث بلغ الانتفاخ 3.938% عند استخدام سوربات البوتاسيوم بنسبة 4% أما كلوريد البوتاسيوم فقد بلغ انتفاخ الصخر الزيتي 11.519% عند نفس التركيز. وقد تم اختيار سوربات البوتاسيوم لأنه مادة صديقة للبيئة وقابلة للتحلل البيولوجي ومن خلال هذه الدراسة تم اختباره لأول مرة في العراق كمثبط لصخور السجيل.

1. Introduction

Evaluating fluid interactions during shale drilling is crucial for addressing wellbore instability issues caused by reactivity and sensitivity of shale [1]. Shale formations, consider up to 75% of drilled intervals, are the primary source of wellbore instability in over 90% of problems [2]. Exposing rocks of clay rich to water causes them to respond and act as unconsolidated rocks. Drilling muds and shale formations interact mechanically and chemically, leading to instabilities. Mechanical interactions are independent of time and depend on the right mud weight. However, chemical interaction is time-dependent [3]. Swelling and reduced wellbore strength can lead to issues such as bit balling, stuck pipe, decreased hole cleaning effectiveness, sloughing and also well loss [4]. The degree of reaction, process, and consequences is depending on the kind of clay. Kaolinite clay disperses in water, whereas illite clay tend to disperses to a lesser amount, Smectite clay tend to swell. This difference in the type and concentration of minerals in the shale rocks leads to the difference in the degree of chemical interaction with water. Drilling shale deposits can cause wellbore instability due to both swelling and dispersion, as well as stress disruption around the well. The borehole stresses are classified into: minimum horizontal stress, vertical stress and maximum horizontal stress [5].

There have been numerous studies conducted to address the problems of instability caused by shale swelling. Oil-Based Muds (OBMs) were the initial alternative proposed as a 'guaranteed' solution to borehole instability induced by shale. Their uses gave an excellent clay inhibition, lubricity, temperature stability and corrosion protection. However, due to environmental issues and laws, OBMs have restricted applicability [6]. This shows the importance of adding different additives to water-based mud (WBM) to enhance its performance, specifically in terms of shale inhibition, through the modification of their density or chemical properties [7].

Research endeavors have been directed towards the identification and the selection of drilling fluid additives with the aim of mitigating swelling and hydration issues connected with active clays and water based muds. These additives, particularly inhibitors are investigated for their potential to address and alleviate the issues [8]. Commonly employed conventional shale inhibitors encompass inorganic salts as potassium chloride (KCl), sodium chloride (NaCl), ammonium chloride (NH4Cl), calcium chloride (CaCl2) and brine electrolyte solutions [9]. Many research has studied unconventional shale inhibitors, including ionic and non-ionic polymers [10]. There is also other additives like soluble silicates, lignosulfonate, amines and polymers. The inhibiting method involves lowering the shale permeability by sealing pores and enhancing the membrane efficiency [11]. Potassium chloride salt is commonly used in water-based muds to suppress reactive shale because of its capacity to replace sodium ions. The capability of clay fixing of K+ ions reduces the shale swelling and lowers the smectite hydration. Because of their tiny size, K+ ions can produce inhibition by fitting into the montmorillonite structure [12].

To formulate an inhibitive water-based mud for a certain shale formation, a sample of the formation must be thoroughly analyzed and characterized. The analysis could examine shale minerals, reactivity, morphology, capillary suction time, fracture development and pore fluid salinity. When selecting the drilling mud additives, the properties that will affect the drilling mud properties must be taken into account (density, viscosity, pH level, filtration volume, lubricity, cost and environmental concerns). After analyzing the shale sample, inhibitors including potassium chloride, silicates, and polyamines can be used. To attain optimal results, it's important to customize the mud for each shale formation due to its unique mineralogy. Creating an inhibitive water-based mud that works well in various kinds of shale is exceedingly difficult [13].

The main purpose of this study is to review the different types of shale rocks, the definition of each of them, and what problems they cause when drilled, in addition to the most important tests that must be performed to reduce the shale problems. Furthermore, the definition of the inhibitors that are used in drilling mud to stabilize the well, as well as list of case studies that contributed in reducing the shale problems with the proposed inhibitors. Finally, this study demonstrates the effect of adding shale inhibitors containing potassium ions as a way to reduce the shale swelling. Potassium sorbate and KCl have been tested to inhibit shale swelling.

1.1. Types of Shale Rocks

Two of the most common types of shale are silty shale and clay shale. Silty shale is predominantly composed of silt-sized particles, which are larger than clay particles but smaller than sand particles. Clay shale is predominantly composed of clay minerals. Clays are classified into five groups:

kaolinite, smectite, illite, vermiculite, and chlorite. The first three kinds are significantly influence on drilling operations. Clay is primarily made up of sheets that include planes of atoms. To avoid contact with water or water-based muds, the clay ought to be neutral with equal numbers of anions and cations. Interlayer components, like isolated or hydrated cations, can neutralize the clay [13]. Clay minerals are typically flaky, mica-like crystalline structure. These flakes are made up of crystal platelets termed unit layers, which may have several octahedral or tetrahedral sheets connected by oxygen atoms. The octahedral sheet comprises six oxygen atoms forming an octahedron with a metal atom in the center. The tetrahedral sheet has a silicon atom in the center of four other atoms, forming a tetrahedron [14].

1.2. Smectite (Montmorillonite)

Montmorillonite, as a member of the smectite family, has a high cation exchange capacity (CEC) which can control the shale hydration that depending on the cation type and pH of the drilling mud. smectite consist of three repeating basis units: a silica tetrahedron, an alumina octahedron, and a silica tetrahedron. The smectite group is characterized by large gaps between the units and the weak bonding, resulting in substantial swelling [15]. To reduce swelling in clays, substitute counter ions like Na^+ and Ca^{++} into the shale. Potassium ions can minimize the swelling phenomena due to their peculiar hydrated size as well as ability to fit within active tetrahedral layer of clay. If potassium ions are transferred into the clay lattice, they are hard to get out, leaving the lattice effectively set. Montmorillonite is normally presented in shallow formations and transitions to illite in deeper intervals. However, the resulting illite may continue exhibit swelling [16].

1.3. Illite

Despite having two tetrahedral silica sheets and a core sheet of alumina octahedral, Illite doesn't hydrate easily in fresh water, unlike montmorillonite. The lack of an expanding lattice prevents water from passing between layers. Powerful interlayer bonds perhaps attributed to the increase in charge near the outer layers of the tetrahedral sheet. The exchange of ion occurs at the external surfaces of illite; however, the increase in volume is lower than that obtained in montmorillonite. Weathering and hydrothermal conditions modify muscovite and feldspar, resulting in illite. The result is that poorly hydrated potassium cations occupy the distance between separate clay crystals, making illite resistant to swelling. However, another kind of illite is created by the transition of smectite in deep layers under high pressure and temperature circumstances. Those multi layers illite may have a greater ability to swell than its initial state [17].

1.4. Chlorite and Kaolinite

Kaolinite, like other clay family members, lacks considerable hydration, yet it can disperse. The mineral is a multilayer silicate composed of a tetrahedral silica sheet and octahedral alumina sheets. Kaolinite exhibits modest swelling and limited cation exchange capacity (1-15 meq/100 g). Aluminum silicates, such as feldspar, undergo chemical weathering to generate this soft and white mineral. Shale with high Kaolinite content can be brittle, leading to concerns about mechanical stability in wellbores [18]. Chlorites are hydrous aluminosilicates made up of an alumina octahedron sandwiched between two silica tetrahedral. Chlorites are phyllosilicate minerals classified into four types based on their chemistry: Clinochlore, Chamosite, Nimite, and Pennantite. Because of their diverse composition, these materials exhibit a wide range of physical and chemical properties and can exist under various temperature and pressure situations [19].

1.5. Vermiculite

minerals of clay, which are hydrated magnesium aluminum iron silicates, are consisting of one sheet of octahedral alumina sandwiched between two tetrahedral silica sheets [20]. vermiculite and Smectite, with high expansively, have a significant influence in shale destabilization. The density and thermal conductivity of vermiculite ensure its mechanical stability and expansion at higher temperatures [21].

2. Mechanism of Clay Swelling

Shale swelling happens when a water molecule covers a clay mineral, raising the distance between sheets, this causes the clay to expand volumetrically. When water molecules come into contact with a clay mineral that has capability to swell, a hydration force is formed, which in turn induces electrical interactions between the clay particles interlayer spacing. The hydrated force is caused by the molecules of water polarity at the Surface that is hydrated and solid, whereas electrical forces are caused by the negative electrical charge on the clay's basal surface [22]. These forces cause the stacked layer to disintegrate. Therefore, the interaction between the drilling mud and the shale formation may cause an increase or reduction in the stress of hydration. Increased hydration stress increases tensile forces, leading to swelling, while decreasing hydration stress decreases tensile forces and make the clay minerals contract, Increasing the effectiveness of inhibition. The type and number of clay minerals in a shale effect its swelling behavior. Clay swelling during drilling may be crystalline or osmotic [23].

2.1. Crystalline Swelling

Clay swelling is caused by interactions between aqueous solutions with varying ion concentrations, including monovalent, divalent, and multivalent ions [24]. The hydration of dry clay's exchangeable cation causes the swelling of crystalline. Initially, the clay contains an excessive negative electric charge on their surfaces. Water molecules shift their negatively charged dipoles heading towards exchangeable cation, causing the clay to be highly susceptible to hydration [25]. This contact causes the clay to expand gradually due to the modest electrostatic force. In the presence of a water-based solution, all clay types experience crystalline swelling, resulting in the formation of single, double, and multiple layers of the cation in the interlayer spaces. The interlayer spacing typically varies from 9 Å to 20 Å [26]. Figure (1) explains the crystalline swelling.

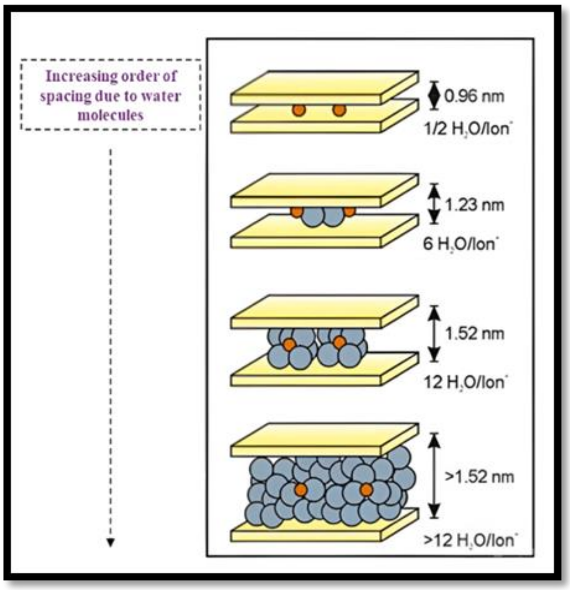


Fig. (1): Crystalline swelling mechanism [27]

2.2. Osmotic Swelling

This phenomenon happens when a cation's interlayer concentration exceeds those of the nearby entire solution due to the difference in salinity. Figure (2) shows that the increasing of the cations concentration throughout clay minerals can cause water to migrate from the surrounding drilling mud into the interlayer voids, resulting in an increasing in the interlayer spacing. Weak van der Waal forces can lead to increase the space of cleave owing to the electrical double-layered effect, producing significant repulsion [28]. Some clay mineral classifications with high exchangeable cations between interlayer spaces are responsible of its swelling without the need for a semi-permeable barrier. Osmotic swelling during drilling operations can cause instability and wellbore collapse if it's not handled promptly [29].

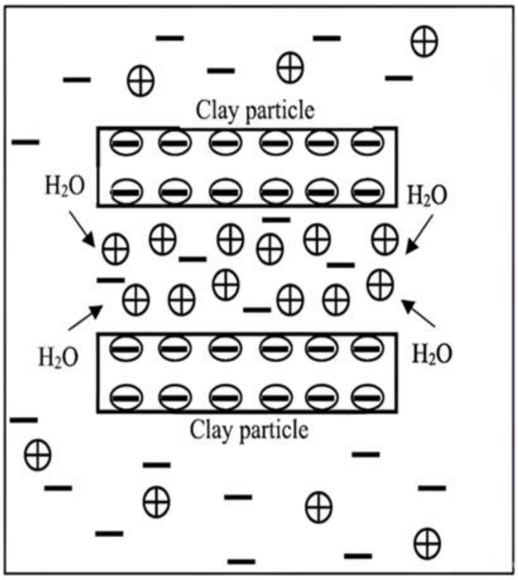


Fig. (2): Osmotic swelling mechanism [30]

3. Review of Mud Treatment

3.1. Oil Based Muds (OBMs)

Replacing water with oil in drilling fluid can help decrease the wellbore instability issues that caused because of hydratable, dispersible, and brittle shale. Oil Based Muds (OBMs) stabilize shale by emulsifying water inside the continuous oil phase, reducing exposure to water to varying degrees. OBMs are the preferred drilling method for 70% of reactive shale deposits, including exploratory and high pressure and high temperature wells [15]. Capillary and polarity effects contribute to the shale stability during OBM drilling. The wettability of oil and shale surfaces require an OBM to withstand hundreds of PSI of capillary pressures. Thus, OBMs is typically caused the shale instability due to the lowering in mud pressure. Even in OBMs, emulsified water can act as a semi permeable membranes, allowing for osmosis through migration into shale. Migration might not happen if the salt concentrations of the mud and shale layers are equal [16]. Another difficulty is selecting an adequate density for OBMs to maintain the mechanical stability of shale. In general, the density decreases as the oil to water ratio increases. On opposite sides, the viscosity increases when the oil to water ratio increases. While OBMs can effectively inhibit shale, they have restricted applicability based on inadequate information gathering, significant mud losses in deep sea drilling, low biodegradable properties cutting contamination, expensive preparation and maintenance expenses, and environmental limits [31].

3.2. Water Based Muds (WBMs)

Three ways are commonly used to address the instability issues caused by interactions between WBMs and shale. They are: ionic inhibition, encapsulation and physical plugging. These treatments can be employed alone or in conjunction with others, depending on the amount and type of clays in the shale. The approached WBM may be less efficient than an OBM, but it may have some uses in reducing shale instability [32]. WBM can help in maintaining the osmotic balance between the drilling fluid and shale, thus reducing the risk of shale swelling. It acts as a clay particle inhibitor, preventing dispersion and swelling of clay minerals in shale formations. Additionally, WBM allows better control of hydraulic pressure in the wellbore, reducing the risk of fluid invasion and shale instability. These benefits contribute to the overall stability and integrity of the shale formation during drilling [33].

- **Ion Inhibition**

Clay chemical stability and dispersion are controlled by exchangeable interlayer cations. Ionic inhibition effectively reduces clay dispersion in WBMs by swapping initial cations with stabilizing cations. The effectiveness of inhibitors is primarily determined by their type and concentration, but hydration may still have an impact [34]. When positioned in water, the clay surface forms strong negative surface charges. Cations in the mud are adsorbed on surfaces due to their attraction force. Ammonium and potassium are the main inhibitive ions while drilling into shale. Their low hydration energies, tiny hydrated diameters, and proximity to the space between montmorillonite and illite layers contribute to their popularity [20].

- **Encapsulation**

The encapsulation mechanism of water-based mud (WBM) involves the formation of a protective barrier around shale particles, preventing direct interaction with the drilling fluid. Long chain polymers like Partially Hydrolyzed Polyacrylamide (PHPA) are commonly used to attach to cutting surfaces, inhibit water absorption and keep cuttings in shape [35]. forming a thin film or coating. This film acts as a physical barrier, inhibiting water penetration and reducing the potential for shale swelling, disintegration, or clay release. The encapsulation mechanism helps maintain the structural integrity of the shale formation during drilling [36].

- **Physical Plugging**

One among the main factors of shale failure is the increase in pore pressure near the wellbore wall.

In permeable rocks, the pressure differential between the drilling mud and pore fluid creates a filter cake that covers the borehole wall, limiting additional mud influx. Shale are nearly impermeable. On this situation, filter cake cannot be formed directly in the formation by the applied mud pressure, resulting in delayed penetration and invasion of mud into the shale[37]. Herein pore pressure becomes greater than the mud pressure, raises the rock stresses around the wellbore, culminating in a massive rock collapse. With these circumstances, encapsulation and ionic inhibition may not be entirely successful, and physical plugging of pore throats might be the most effective option [38]. Thermally activated polyglycol is possibly the best solution at these conditions. Their compounds are often stable in drilling fluids at the surface. When the temperature reaches a critical threshold, the polyglycol forms hydrophilic and hydrophobic phases. The transition temperature is known as the cloud point, and it can be controlled using certain polyglycols and mud salinity modifications. The generated micro-gels are large enough to clog the shale pore throats, preventing pressure transmission. However, various additions can impact the cloud point transition. The precise cloud point must be carefully developed in the laboratory, and the drilling fluid must contain an adequate concentration of glycol. The glycol concentration should also be checked in the field because it depletes with time. Asphaltenes and Gilsonite are two other alternatives for blocking the pore throats of shale and their microfractures. Combining them with appropriate Loss Circulation Materials (LCM) can limit filter loss into fractures [39].

4. Review of Shale Inhibition Additives

To improve shale inhibition, water-based drilling fluids and completion brines may contain inorganic or organic additions, typically salts. Potassium chloride is a popular inorganic shale inhibitor, while quaternary amine salts are preferred organic shale inhibitors. There are two types of shale inhibitors: temporary (which is additionally referred to as clay-controlling additives) and permanent (also described as clay stabilizers). Both groups of shale inhibitors have advantages and disadvantages [15].

4.1. Inorganic Shale Inhibitors

calcium chloride, Sodium chloride, and potassium chloride are clay controlling chemicals that temporarily inhibit shale. These salts are only useful when its used in water-based drilling fluids that come into contact with clays. When the salts are exhausted or replaced by new water, the clay hydrates and swells, causing the drilled formation to become unstable. Drilling fluids usually contain salt concentrations ranging from 2 to 37% [11]. Salts reduce clay swelling through a number of processes. The cation exchange reaction limits the quantity of water absorbed by hydratable cations on the surface of expanding clays. This method is unsuccessful in clays that lack exchangeable cations. To prevent osmotic hydration, heavy brines or electrolytes can be added to enhance water phase ionic concentration. This type of shale inhibition is extremely successful in shale formations with Smectite as the primary clay mineral [40]. Inorganic salts are reasonably affordable and widely available around the world. They are chemically stable and can be used in various drilling situations, including high temperatures and pressures, as well as a large range of pH values. However, in high concentrations, these salts may harm chemical and biological ecosystems. Above specific limits, these salts can negatively impact water and soil quality for agriculture. High salt concentrations in drilling fluids have made them unsuitable for environmentally sensitive land-based drilling locations, resulting in surface disposal limitations from environmental authorities. In exploratory drilling, high conductivity from these salts can affect the sensitivity of induction logs [41]. These salts limit the flexibility of drilling fluid mixtures and are incompatible with other additives. For many years, potassium salt has been a reliable shale inhibitor, whether used alone or in addition with other inhibitors [42].

4.2. Organic Shale Inhibitors

Different cationic sources have been investigated in literature to address the environmental issue and to figure out its performance with salts, specifically potassium chloride. This led to the creation of novel cationic amine chemicals as shale inhibitors. These shale inhibitors can be divided as more persistent shale inhibitors, sometimes known as clay-stabilizers [43]. The interaction between shale inhibitors and shale is by cation exchange mechanisms, whether within the matrix or on its surface. These amine-based shale inhibitors are highly effective for shale with strong cationic exchange capability. Several cationic organic amine salts have been utilized in the field; however, they were unstable at high pH and temperatures [44]. When amine salts decompose, they emit a strong ammonia odor and lose their effectiveness as shale inhibitors. Exotic and quaternary amines have been formed. to address toxicity, odor, stability, and performance issues with simple amine salts. Amine shale inhibitors can be categorized into three types based on their structure and chemistry: monomeric, oligomeric, and polymeric [45].

4.3. Polyamine Shale Inhibitors

Polymeric amine shale inhibitors are considered to be more durable shale stabilizers. significant molecular shape exhibited by polymeric cationic amines cannot penetrate clay layers as well as oligomeric quaternary amines. therefore, adsorption happens mostly at the surface of the clay, resulting in a lower efficient inhibitor of shale in strongly swelling clays [46]. Oligomeric cationic amines exhibit superior inhibition capabilities against shale formations compared to a multitude of cationic sites. Conversely, polymeric cationic amines present certain drawbacks, including compatibility issues with anionic drilling fluid additives, as well as elevated viscosity and toxicity in drilling fluid formulations. Nevertheless, the industry has witnessed the production and application of numerous polymeric cationic amines [47].

5. Shale characterization techniques

5.1. Dispersion Test (DT)

The test determines the tendency of dispersion of a shale samples following exposure to drilling mud. The test involves rolling a predetermined quantity of shale cuttings in a hot-rolling cell with the drilling fluid to conduct the test. After 16 hours of hot-rolling at 150 °C, the cuttings of shale are collected on a screen, washed, and dried in an oven at 105 °C. The cuttings is then weighted to calculate the percentage of cuttings recovery [48]. Results are reported as a cuttings percentage recovered. A high cuttings recovery percentage is an indicator of the effective resistance against dispersion, as a significant portion of the cuttings remain intact [49].

5.2. Linear Swelling test (LST)

This test measures the swelling propensity of shale samples after exposure to drilling fluids. To conduct this test, place an outcrop or natural shale sample of particular dimensions or a reconstituted shale pellet into a linear swelling meter and fill with drilling fluid. When the shale pellet makes contact with the drilling fluid, it begins to swell. Swelling is assessed as an increase in sample volume or linear expansion. The measurements are normally presenting in a plot of swelling % over time. A high ultimate swelling percentage indicates an inadequate drilling fluid resistance to swelling [50].

5.3. Scanning Electron Microscope (SEM)

Scanning electron microscopes give high-resolution, magnified views of shale structures. This method provides a more accurate 3-D study of micro cracks and pores than a transmitted light microscope for thin section analysis. Combining an energy-dispersive X-Ray fluorescence detector (EDAX) with a scanning electron microscope (SEM) enhances sample analysis by revealing chemical composition. The combination of SEM and EDAX can identify the composition, size, and shape of pores, mineral content, and embedded minerals in the sample matrix [51].

5.4. Cation Exchange capacity (CEC)

The CEC test analyzes the cations that can be exchanged on the clay surface. particles of clay contain negative surface charges that can be balanced by exchangeable cations with positive surface charges, such as Ca2+, Na+, K+ and Mg2+. The CEC measurement follows the API's approved methylene blue (MB) capacity test [52]. In this test, finely ground shale is dispersed in water with a little amount of dispersant, sulfuric acid, and hydrogen peroxide. To titrate, boil the mixture, cool it to room temperature, then add methylene blue solution dropwise. To attain the titration endpoint, deposit a drop of the combined sample on filter paper, resulting in a faint blue halo around the dyed solids [53]. The CEC is measured in milliequivalents per 100g of clay (meq/100g), with reactive clays typically exceeding 20 meq/100g and moderately reactive clays ranging from 10 to 20 meq. Smectite typically has a CEC of 80-120 meq/100g [54].

5.5. Capillary Suction Time (CST)

This test evaluates the time required for a clay/shale slurry to move a specific distance. Each test requires 3 g of dry shale material. Shale dispersion in the inhibitive fluid can lead to lowering the CST magnitudes because of flocculation. In contrast, reactive shale with high smectite clay concentration typically have higher CST values [55].

5.6. X-Ray Diffraction (XRD)

X-Ray diffraction (XRD) is a standard method for identifying minerals in shale composition. The equipment rotates the shale sample while simultaneously hitting it with x-ray rays. Minerals' crystalline structure diffracts the x-ray beam, resulting in similar characteristics observed by the equipment. XRD can be combined with clay fraction analysis to better quantify clay minerals in bulk samples [56].

5.7. X-Ray Fluorescence (XRF)

X-ray fluorescence (XRF) testing is not a damaging analytical technique for determining the elemental composition of a substance. In the context of shale characterization, XRF testing is critical for understanding the mineralogy, elemental composition, and physical properties. It aids in appraising shale's potential as a hydrocarbon source, determining its appropriateness for various purposes, and investigating its geochemical properties. By measuring components in shale samples, XRF analysis gives vital information about lithology, porosity, permeability, mechanical strength, source rock potential, thermal maturity, and depositional environment. This information helps with petroleum exploration, geotechnical engineering, and environmental assessments. Overall, XRF testing is a significant technique for shale characterization, providing useful information for decision-making and research [57].

Table (1) reviews the results of some researchers and the methods that were used for the purpose of testing chemicals to reduce the interaction of shale with drilling mud. Realize the importance of various inhibitors in reducing the shale reaction, which leads to reducing the problems associated with drilling these formations.

Table (1): A selection of literature on characterization techniques used in shale inhibition experiments

Reference	Type of Shale	Technical Characterization of Shale	Type of inhibitor with its concentration wt%	Swelling results %	Desperation results %
[58]	Montmorillonite	LST, XRD, CST	Drilling mud	120	
			3%KCl	112	
			3%K- sorbate	80	
			6%KCl	87	
			6%K- sorbate	70	
[59]	illite. Chlorite and Kaolinite	HRT, ZPT, SEM, LSM	Drilling mud	6	25
			2% SH-SiO2	1.5	60
			2%KCl	4	30
			3% SH-SiO2	1	75
			3%KCl	3.5	35
[60]	Montmorillonite, kaolinite	LST	Drilling mud	65	
			3% K-citrate	35	
[61]	Montmorillonite	LST, HRT, XRD, SEM	Drilling mud	10	17
			4% Polyether amino	9	96
			4% KCl	9	25
[62]	smectite and sodium Montmorillonite	HRT, SEM	Drilling mud		36
			3% GGRE		61
			3% KCl		49
[63]	Montmorillonite, kaolinite and illite	ZPT, HRT	Drilling mud		30
			3.5% (CO)		41.5
			3.5% KCl		61
[64]	Montmorillonite	ZPT, WAT, LST, HRT	Drilling mud	90	43
			2% tallow amine ethoxylate	80	69
			2% KCl	83	71
[65]	Montmorillonite, kaolinite	LST, ZPT,SEM, WAT, CST	Drilling mud	30	
			2% Quaternary ammonium gemini surfactants	20	
			2% Na-silicate	17	
			2% KCl	23	
[66]	Montmorillonite	ZPT, LST	Drilling mud	120	
			0.5% synthesized carboxybetaine zwitterionic surfactant	70	
[67]	kaolinite, illite	LST, XRD	Drilling mud	46	

			4% Novel Polymeric	31
[68]	Montmorillonite, kaolinite	LST, CST, ZPT	Drilling mud	160
			3%NaCl	85
			3%CaCl2	90
			3%KCl	80
			7% NaCl	80
			7% CaCl2	85
			7%KCl	75
[69]	smectite, illite, kaolinite ,chlorite	CEC, CST, XRD, XRF, LST	Drilling mud	30
			2%AD-Glycol with 7%KCl	16
			3% AD-Glycol with 7% KCl	15
			3% with 10% KCl	14
			2% AD Polyamine with 3%KCl	13.8
[70]	kaolinite, illite , chlorite	XRD, XRF, LST	Drilling mud	26
			5%Polyamine	25
			5%KCl	13.5

6. Material and methods

In this study, XRD and XRF tests were used to identify the type of shale and its mineral composition. To evidence the importance of adding inhibitors for reducing the shale problems encountered during drilling operations, the linear swelling test (LST) was also used to know the effect of 4% of potassium sorbate and KCl on the swelling of the shale. To prepare the shale samples for testing, they were washed to rid them of drilling mud and then dried for two hours at a temperature of 105 C. As for preparing the drilling mud, 22.5 g of bentonite was added to 350 ml of distilled water, mixed for 20 minutes, left for 24 hours, and then a concentration of 4% (14 g) of potassium sorbate and KCl was added.

6.1. XRD

1 gram of shale was taken, ground slightly, and placed in the XRD device (Figure 3) for 10 minutes to obtain the results. The diffraction pattern reveals the nature of shale cuttings. Figure (4) shows the results of the XRD test, where through the diffraction pattern it is clear that the type of the shale samples is kaolinite and it’s also contained quartz and calcite.

Fig. (3): X-ray Diffraction (XRD Model Shimadzo 600)

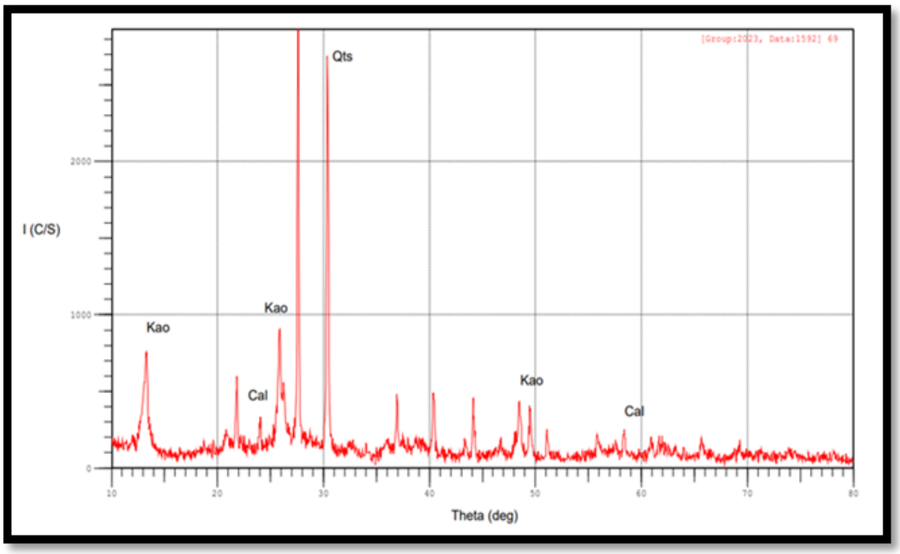


Fig. (4): XRD result of shale sample

6.2. XRF

The XRF test requires 5 grams of shale, ground well and pressed with 5 tons of pressure to made pellets (Figure 5). then placed in an XRF (model: Spectro Xepos) device (Figure 6) for 20 minutes to obtain the percentages of mineral composition contained in the shale samples. Table (2) shows the percentages of minerals obtained from the XRF test, as the table shows the highest percentages of minerals found in shale samples, which are sodium, magnesium, aluminum, silicon, sulfur, potassium, and calcium with other elements.

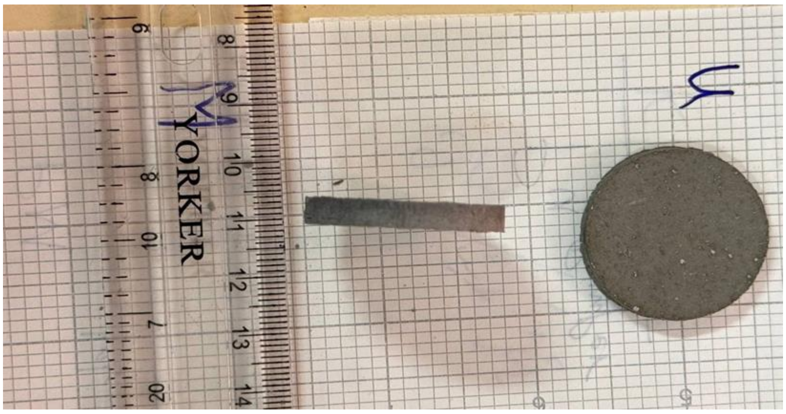


Fig. (5): Pellets Samples Preparation from Shale Powder for XRF test

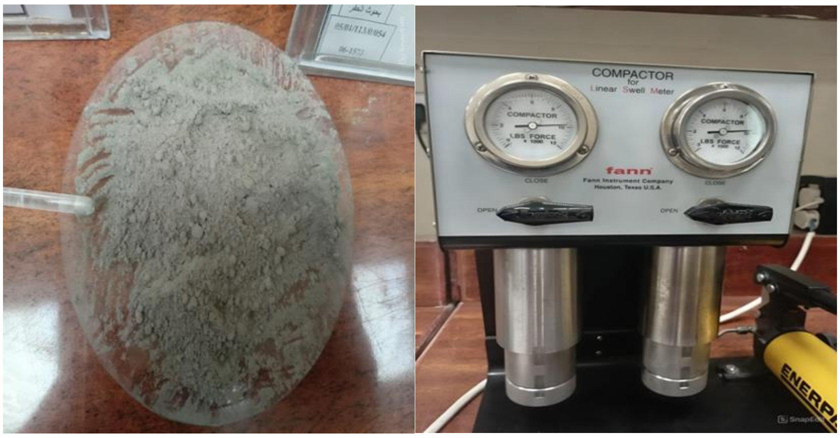
Fig. (6): X-ray Fluorescence (XRF Model Spectro Xepos)

Table (2): Mineral concentration of shale sample obtained from XRF test

symbol	Element	Concentration %
Na2O	sodium	1.051
MgO	magnesium	1.68
Al2O3	aluminum	14.19
SiO2	silicon	45.17
SO3	sulfur	2.38
K2O	potassium	1.335
CaO	calcium	12.56

6.3. LST

The experimental procedure involves the preparation of shale samples by initially grinding them into fine powders. Subsequently, 25 grams of shale are subjected to a high-pressure into compactor utilizing a force of 10,000 lbs for one and a half hours (Figure 7), resulting in the formation of compact plugs (Figure 8). These plugs are subsequently placed within a linear swelling meter device and immersed in drilling mud (Figure 9). The swelling behavior of the plugs is then monitored and recorded over a three-hour time period. When the shale is interacted with drilling mud without any additives (only bentonite), the shale swelling reaches to higher value which is 16% (Figure 10). By testing potassium sorbate and KCl at a concentration of 4%, the results showed that potassium sorbate caused less shale swelling than KCl. The LST results of Figure (7) showed that potassium sorbate can reduce the shale swelling to be only 3.938%, while KCl reduced the shale swelling to be 11.519%. This shows the better effectiveness of potassium sorbate than KCl in mitigating the shale swelling, as this substance is considered environmentally friendly and biodegradable, which makes it a suitable alternative to KCl.



(a) (b)

Fig. (7): a) 25 g of Shale Cutting, b) LSM Compactor

Fig. (8): Shale Plugs Results

Fig. (9): Linear Swelling Meter (LSM)

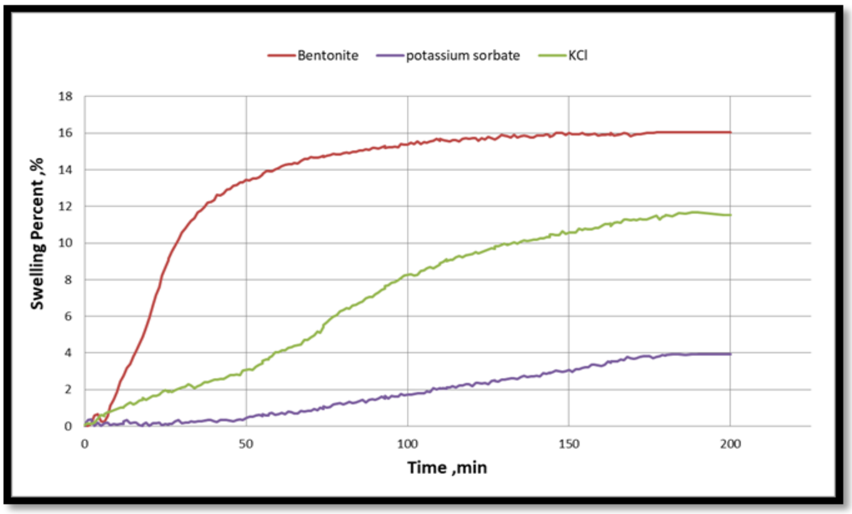


Fig. (10): Linear Swelling Meter results of 4% concentration

7. Conclusions

Shale formations have been identified for their high clay concentration, which can cause many drilling problems such as wellbore instability, blocked pipe incidents, thus decreasing the drilling efficiency. To overcome this challenge, on the other hand, inhibitors play an important role in minimizing the issues connected with such shale problems during drilling operations. This study demonstrates the review of using different inhibitors to reduce the chemical interaction of shale with drilling mud. An experimental work to investigate the efficiency of potassium sorbate and KCl in reducing the shale swelling was also performed. The key findings of this study can be summarized as follow:

1. Shale swelling occurs when water molecules interact with clay minerals, leading to an increase in the distance between clay sheets and volumetric expansion of the clay. When exposed to water, clay-rich rocks in shale formations behave like unconsolidated rocks.

2. Wellbore instability caused by shale swelling and due to a reduction in rock strength can lead to various drilling issues, including stuck pipe and decreased hole cleaning effectiveness.
3. Effective drilling fluid treatments play a crucial role in mitigating adverse interactions with shale formations. By focusing on strategies to prevent shale swelling, dispersion, and other related issues, operators can enhance wellbore stability and optimize drilling efficiency.
4. It is necessary to know the type of shale that is being dealt with by conducting tests to determine the chemical composition of the shale and how it interacts with water, and thus choosing the appropriate inhibitor.
5. Calcium chloride, sodium chloride and potassium chloride are inorganic salts commonly used as temporary shale swelling inhibitors in water-based drilling fluids through different processes such as cation exchange reactions and osmotic hydration prevention.
6. It is also important to consider the choice of shale inhibitors in terms of their impact on the environment. In the study, potassium sorbate was used as an alternative to KCl due to the presence of chlorine, which is considered non-degradable and polluting to the environment.
7. The results of this study also showed that potassium sorbate at a concentration of 4% can reduce the shale swelling to 3.938%, while KCl at the same concentration reduces the shale swelling to 11.519%. Potassium sorbate is more effective and environmentally friendly, making it a suitable alternative to KCl.
8. Selecting different shale inhibitors is primarily depending on the shale types and characteristics and how the addition of these materials can mitigate the shale swelling.
9. Overall, the use of shale inhibitors represents a valuable approach to overcoming shale-related issues in drilling operations. By incorporating these inhibitors into drilling fluid treatments, operators can effectively manage shale interactions, reduce risks, and achieve greater success in their drilling endeavors.

Author Contributions Statement: Elaf Y. Fadhil contributed to the Conception; Methodology; Investigation/ Experiments; Modeling/ Simulation; Writing – Review & Editing. Farqad A. Hadi contributed to the Conception; Methodology; Investigation/ Experiments; Modeling/ Simulation; Writing – Original Draft; Writing – Review & Editing. Ali N. Al-Hasnawi contributed to the Conception; Investigation/ Experiments; Modeling/ Simulation; Writing Original Draft; Writing – Review & Editing. All authors have read and approved the final version of the manuscript.

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