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Abstract

Excess water production during oil production is a big challenge that may affect the economics and environmental concerns. Different methods have been developed to reduce water production and improve the efficiency of oil recovery. One promising approach is using a nanocomposite that creates a gel for plugging fractures in the reservoir. This system can potentially improve volumetric sweep efficiency during oil production by decreasing water permeability and creating a better path for the oil to flow to the well.

This study investigates the potential of an ECO-clay system comprising Laponite and Hydrolyzed Polyacrylamide (HPAM) as a water conformance technique in fractured reservoirs. The investigation involves bulk experiments to screen different concentrations of clay and polymer, while also evaluating the effects of various ions and additives on gel performance. Promising systems are subsequently subjected to filtration tests to assess their suitability for gel placement into fractures. Ultimately, the goal was to identify the most effective gel system and conduct core flooding tests to evaluate its potential for plugging fractures and improving oil recovery.

Among the systems tested, two were selected for dynamic experiments: A Laponite S482-CaCl₂ system and a nanocomposite system with HPAM addition. Both showed good results in the filtrations and floods, achieving a water permeability reduction of 19.6 and 5.3 times lower than the pre-treatment permeability respectively. Finally, the Laponite S482-CaCl₂ system represented the most promising gel system for fracture plugging in chalk, because its higher permeability reduction.

These findings highlight the potential of the identified gel system for effectively addressing water production issues in fractured chalk reservoirs, thereby improving oil recovery efficiency, reducing water production and CO₂ emission. To improve the understanding and implementation of these gel systems, further work is required in a more in-depth gel structure tests, long-term stability and performance evaluation, compatibility with other reservoir fluid study, and finally field-scale experiments and simulations. Addressing these issues will help create a reliable and cost-effective fractured reservoir water conformance technique.

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Nomenclature

EOR	→	Enhanced Oil Recovery
F_{rrw}	→	Residual Resistant Factor
G/W	→	Gas & Water
HPAM	→	Hydrolyzed Polyacrylamide
k	→	Permeability
M	→	Mobility Ratio
N	→	Nanocomposite
O/G/W	→	Oil & Gas & Water
O/W	→	Oil & Water
u	→	Flow rate
WOR	→	Water Oil Rate
λ_o	→	Oil Mobility
λ_w	→	Water Mobility
μ	→	Viscosity
∇P	→	Pressure Gradient

1. Introduction

1.1. Motivation

Excess water production is a major issue that reduces the amounts of hydrocarbons recovered, increases production costs, and creates environmental concerns. It can even result in early well abandonment and leave unrecoverable hydrocarbon in mature oil fields (Goudarzi et al., 2015). To address these problems, it is crucial to use methods that reduce water production and improve volumetric sweep efficiency in low permeable oil zones (Seidy Esfahlan et al., 2021). By reducing water production, operators can maintain the field's profitability, minimize the need for water handling and disposal facilities, and reduce the environmental impact of their operations.

The management and control of water production is referred to as water conformance. It typically involves a combination of techniques such as chemical treatment (Abdel-Basset et al., 2020; Guo et al., 2022; Hernandez et al., 2016; Luo et al., 2020; Rajabi et al., 2023), where a chemical is injected into the well to reduce the permeability of high permeable zones; mechanical isolation (Abdel-Basset et al., 2019; Abdel-Basset et al., 2020; Mohamed et al., 2015; Qayed Subaihi et al., 2019), which physically blocks permeable zones using devices like casing, packers, or screens; intelligent completion (Abdel Rafea & Criado, 2018; Ismail et al., 2019; Masoudi et al., 2015; Vieira et al., 2020), where sensors and control systems are used to monitor and manage fluid flow in a well; profile modification (Guest et al., 2019; Ismail et al., 2019), by changing the well profile or completion design; and gel-based water conformance (Bai et al., 2022; Goudarzi et al., 2015; Seidy Esfahlan et al., 2021), which uses gels to control fluid flow in a well. The goal of these techniques is to prevent or reduce water entering the well and improve production efficiency, as can be observed in the figure 1.

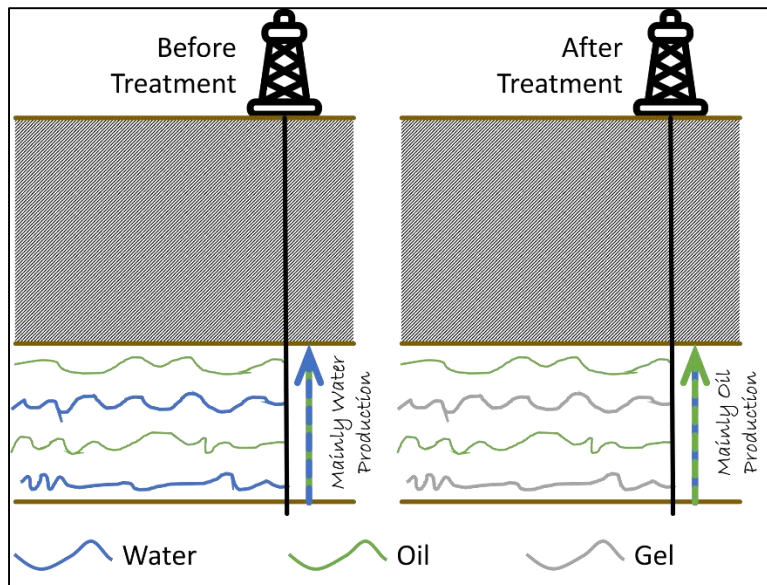


Figure 1. Water conformance with gels for plugging fractures in reservoir. **Left image:** Before fracture, many fractures where water flows easily to the well, thus mainly water production. **Right Image:** After Treatment, fractures plugged, better path for the oil to the well, thus mainly oil production.

There are several types of gels that are being explored as options for water conformance techniques in the oil and gas industry (Aldhaheri et al., 2021; Baloochestanzadeh et al., 2021; Ghriga et al., 2019; Heidari et al., 2022; Seidy-Esfahlan et al., 2022). Nanocomposite gels are a promising option for water conformance in the oil and gas industry, offering advantages over other types of gels. Unlike traditional polymer gels, nanocomposite gels are created by adding nanoparticles to the gel mixture, which improves their viscosity and reversibility (Almohsin et al., 2014; Bai et al., 2018; Chauhan et al., 2017; Jia, Yang, et al., 2020; Thakur & Kessler, 2015; Tongwa & Baojun, 2015; Yang et al., 2023). In addition, they are generally considered to be less toxic than other types of gels (Hatzignatiou et al., 2016; Jia, Yang, et al., 2020; Yudhowijoyo et al., 2019). These properties make them well-suited for controlling fluid flow in oil and gas reservoirs and reducing the production of unwanted water.

In recent years, clay/polymer nanocomposite gels have gained attention for their potential to plug fractures and control water shutoff in reservoirs (Asadizadeh et al., 2021; Azzam, 2012; Singh & Mahto, 2016, 2017; Singh et al., 2018). The clays used in these gels typically include smectite clays like montmorillonite (Thakur & Kessler, 2015) and bentonite (Aguiar et al., 2020), or synthetic clays like Laponite (Aalaie & Youssefi, 2012). Of these, Laponite is particularly appealing for use in nanocomposite gels due to its high surface area, clear gel-forming ability, and stability in the presence of certain polymers (Du et al., 2022; Jia, Xie, et al., 2020; Jia, Yang, et al., 2020).

1.2. Objectives

The primary objective of this thesis is to investigate the feasibility of using Laponite and HPAM to make a nanocomposite gel for plugging fractures in chalk reservoirs. Furthermore, this study aims to qualitatively assess the efficacy of these nanocomposite hydrogels in preventing water flow within fractured reservoirs.

Secondary objectives include:

- To determine the effect of varying the concentrations of Laponite and HPAM on the rigidity and gel time of the resulting gelant, as well as its sensitivity to temperature and cations.
- To perform a screening to obtain the most promising gel systems, checking their feasibility to pump it into a chalk fracture.
- To evaluate the performance of the gel systems in preventing water flow during flooding tests and identify the most effective formulation for potential application in fractured reservoirs.

2. Background

2.1. Introduction to Water Management in Oil and Gas Industry

The oil and gas industry is a crucial component of the global energy supply chain, providing the energy needed to power homes, businesses, and transportation systems around the world (IEA, 2020). However, the production of oil and gas is often accompanied by the unwanted production of large volumes of water. This excessive water production presents significant challenges to the industry, including increased costs, decreased efficiency, and potential environmental risks (Seright & Brattekas, 2021). To address these challenges, water management has become a critical focus for the industry.

2.1.1. Excessive Water Production

Excessive water production is a significant and complex challenge faced by the oil and gas industry, particularly in mature fields (Mahgoup & Khair, 2015). It can have serious economic and environmental consequences, impacting both hydrocarbon recovery efficiency and production costs. For example, when natural or injection water flows into the reservoir, there is a risk that, instead of sweeping through the targeted zones and effectively recovering oil, the water mix with the hydrocarbons or possibly flows faster than them, resulting in a lower recovery efficiency (Du et al., 2005; Moradi et al., 2018).

Table 1. Excess Water Production Problems and Treatment Categories (Categories are listed in increasing order of treatment difficulty). Taken from (Lane et al., 2003).

Treatment Categories	Excess Water Production Problems
A “Conventional” Treatments Are Normally an Effective Choice	<ul style="list-style-type: none"> • Casing leaks without flow restrictions • Flow behind pipe without flow restrictions • Unfractured wells (injectors or producers) with effective barriers to crossflow
B Treatments with Gelants Normally Are an Effective Choice	<ul style="list-style-type: none"> • Casing leaks with flow restrictions • Flow behind pipe with flow restrictions • “2D coning” through a hydraulic fracture from an aquifer • Natural fracture system leading to an aquifer
C Treatments with Preformed Gels Are an Effective Choice	<ul style="list-style-type: none"> • Faults or fractures crossing a deviated or horizontal well • Single fracture causing channeling between wells • Natural fracture system allowing channeling between wells
D Difficult Problems: Gel Treatments Should Not Be Used	<ul style="list-style-type: none"> • Three-dimensional coning • Cusping • Channeling through strata (no fractures), with crossflow

In some cases, the amount of water produced can be so high that it becomes economically or technically unfeasible to separate the water from the oil and gas, leading to a reduction in overall well productivity (Fakhru’l-Razi et al., 2009). In fact, according to industry reports,

water production can account for up to 97% of the total fluid produced in some reservoirs, highlighting the urgent need for effective water management techniques (Sachica Avila et al., 2020).

To effectively address excess water production in the oil and gas industry, it is crucial to identify the specific problem and tailor a solution accordingly. There are various factors that can cause excess water production, and each requires a unique approach. For optimal results, it is essential to correctly identify the nature of the issue at hand. Table 1 presents four categories of water production problems, listed in ascending order of treatment difficulty (Lane et al., 2003). However, the degree of treatment difficulty can vary within each category, so the order is only a rough guide.

2.1.2. Existing Water Conformance Techniques

To address the problem of excessive water production in oil and gas reservoirs, various water conformance mechanisms have been developed and employed over the years. This concept refers to all the techniques and procedures utilized to stop excessive and harmful water production (Imqam, 2015; Robert D. Sydansk, 2011). The treatments are typically divided in mechanical, completion and chemical solutions (Abdel-Basset et al., 2020; Joseph & Ajiienka, 2010; Masoudi et al., 2015; Xindi & Baojun, 2017).

Mechanical Solutions

Mechanical solutions involve physically blocking or diverting water flow to prevent it from interfering with hydrocarbon production (Ortiz Polo et al., 2004). Some examples of mechanical solutions include setting packers (Xue & Yang, 2007), which are inflatable devices that can be used to isolate different zones within a wellbore, and setting plugs or bridge plugs to block off zones that are producing excess water. Gravel packing can also be used to physically block water flow into the wellbore while still allowing hydrocarbons to flow through (Marshall et al., 2000). Mechanical solutions can be effective in certain situations, but they can also be costly and difficult to implement, especially in older wells with complex wellbore geometries (Joseph & Ajiienka, 2010; Xindi & Baojun, 2017).

Completion Solutions

Completion solutions are designed to control water production by modifying the completion design of a well (Dani et al., 2022). These solutions aim to create a barrier between the water and oil zones, preventing the water from entering the wellbore. One common completion technique is to use a packer to isolate the producing zone from the water zone (Ueta et al., 2008). This packer can be used in conjunction with screens or liners to control water flow into the well. Another approach is to use intelligent completion technology, such as inflow control devices or downhole sensors, to monitor and regulate water and oil flow in the well (Al-Anazi et al., 2016; Das & Al-Enezi, 2014; Shahreyar et al., 2020).

These technologies can help optimize production by selectively controlling the inflow of water and oil, thus minimizing water production and maximizing hydrocarbon recovery. However, these completion solutions can be expensive and may require significant upfront investment (Ramizah et al., 2017).

Chemical Solutions

Chemical solutions involve the injection of various chemical agents to control water production (Wiggett, 2014). One of the most commonly used chemical agents is polymer gel (Bai et al., 2022; Sagbana & Abushaikha, 2021), which can be used to reduce permeability in high water-cut zones, effectively diverting water and increasing hydrocarbon production. Other chemical solutions include foam (El-Karsani et al., 2014; Wassmuth & Singhal, 1997), which is injected to reduce gas mobility and improve sweep efficiency, and surfactants (Chen et al., 2014) can be used to lower interfacial tension between oil and water, thereby reducing the tendency of water to flow towards the wellbore.

Chemical solutions are often used in combination with mechanical or completion solutions to achieve optimal results (Joseph & Ajiienka, 2010). However, the use of chemical solutions can be costly and requires careful consideration of potential environmental impacts (Hill et al., 2012).

2.2. Flow in a Porous Media

Flow in a porous media is a complex phenomenon that is relevant to many areas of engineering and science, including petroleum engineering (Vafai, 2015). When a fluid (such as oil, gas, or water) flows through a porous media (such as rock or sand), it encounters resistance due to the complex geometry of the media.

One way to describe flow in a porous media is by using Darcy's Law, which states that the rate of flow through a porous media (u) is proportional to the pressure gradient (∇P) and the permeability of the media (k), but inversely to the viscosity of the fluid (μ) (Bedeaux et al., 2020), as equation 1 shows.

Equation 1. Darcy's Law Equation

$$u = -\frac{k}{\mu}\nabla P$$

Darcy took into account the porous media properties and the fluid properties when he included k and μ in the equation. Permeability is a measure of how easily fluids can flow through the media determined by the size, shape, and connectivity of the pores. On the other hand, viscosity is a measure of a fluid's resistance to flow that depends on its internal forces, temperature and pressure. Finally, the pressure gradient represents the driving force that causes fluid to flow through the porous media from the highest to the lower pressure.

This is a key concept for understanding how oil and gas are produced from reservoirs. Understanding the properties of the reservoir rock and the fluids that are present is crucial for predicting the behavior of the reservoir and designing effective production strategies (Sheikholeslami & Shehzad, 2018).

The behavior of fluids in porous media can also be affected by factors such as wettability, capillary pressure, and relative permeability. Wettability can influence the distribution of fluids in the reservoir, which is the tendency of a fluid to either wet or not wet the solid surface of the porous media (Moldoveanu & David, 2017). Capillary pressure describes the pressure difference between two phases (such as oil and water) at the interface between them, and can cause fluids to be trapped in small pores (McPhee et al., 2015). Relative permeability indicates the permeability of a fluid phase in the presence of another fluid phase, and can

affect the efficiency of fluid displacement during production operations (Gupta & Yan, 2016).

Understanding the flow behavior in porous media is essential for reservoir simulation, which is a tool used to predict the performance of an oil or gas reservoir over time. Reservoir simulation involves the construction of a numerical model of the reservoir and the simulation of fluid flow and transport in the model. The accuracy of the simulation depends on the accuracy of the input parameters, such as the rock and fluid properties, which are related to the flow behavior in porous media (Begum et al., 2022).

Fluid flow in porous media can be classified into two types: single-phase flow and multiphase flow. Single-phase flow refers to the flow of a single fluid phase (such as oil, gas or water) through the porous media, while multiphase flow refers to the flow of two or more fluid phases (such as O/W, G/W or O/G/W) through the porous media. Multiphase flow is more complex than single-phase flow, as it involves interactions between the different fluid phases and can result in phenomena such as fluid trapping and saturation changes (Takacs, 2015).

2.2.1. Mobility Ratio

The determination of fluid phase mobility within a porous medium is established through utilization of the Darcy equation. The absolute permeability of the porous medium is denoted by k_i in the context of single-phase flow. The effective permeability of a flowing phase in multiphase flow is dependent on the saturation of the phase. The expression for the mobility of the fluid phase is denoted by λ_i (Green & Willhite, 2018).

$$\lambda_1 = (k_1/\mu_1)$$

The mobility ratio, denoted as M , is a valuable concept in calculations related to displacement processes, which pertains to the relative mobility of the fluid phases involved in the displacement. For a water flood operation, in an oil reservoir, it can be written as shown in the next equation (Green & Willhite, 2018)

$$M = \lambda_w/\lambda_o = (k_{rw}/\mu_w)(\mu_o/k_{ro})$$

Where λ_w and λ_o are the water and oil mobilities, respectively, K_{rw} and K_{ro} are respectively the relative water and relative oil permeabilities, μ_w and μ_o are the water and oil viscosities.

The mobility ratio is a significant parameter that serves as an indicator of the displacement process's state. The decrease in sweep is observed in both areal and vertical dimensions as the value of M increases, given a constant volume of injected fluid (Green & Willhite, 2018). The mobility ratio serves as a reliable metric for assessing the stability of a displacement process. When the mobility ratio (M) exceeds 1, it is possible for viscous fingers to emerge during a water-oil flooding process, leading to the displacement of oil by water. On the other hand, a mobility ratio that is lower than 1 ($M < 1$) is considered advantageous as it signifies a displacement front that is stable.

2.2.2. Residual Resistance Factor

The residual resistance factor (F_{rr}) can be mathematically expressed as the ratio of a fluid mobility before polymer/gel injection to the ratio of the fluid mobility after polymer/gel injection. The aforementioned parameter pertains to the extent of reduction in permeability resulting from the introduction of polymer or gel during injection.

$$F_{rr} = \lambda_i / \lambda_{ig}$$

Where λ_i is the mobility of a fluid (water or oil) before polymer/gel injection and λ_{ig} is the mobility of the same fluid after polymer/gel injection. Considering water as the working fluid, and taking into account that the fluid is the same without changes in its conditions, thus viscosity remains equal. The equation can be written

$$F_{rr} = k_w / k_{wg}$$

Where k_w is the mobility of a fluid (water or oil) before polymer/gel injection and k_{wg} is the mobility of the same fluid after polymer/gel injection. An effective modifier for relative permeability should possess a high residual resistance factor for water, while concurrently exhibiting a low residual resistance factor for oil.

2.2.3. Different Rock Types

The properties and characteristics of reservoir rock types, such as permeability, porosity, and mineralogy, can have significant impacts on fluid flow and transport properties in reservoirs. While sedimentary rocks such as sandstones and carbonates are the most commonly encountered reservoir rocks (Dandekar, 2013), there are instances where hydrocarbons are found in highly fractured igneous and metamorphic rocks (Ozdemir & Palabiyik, 2022; Schutter, 2003). These occurrences are, however, relatively rare and on a much smaller scale compared to those found in sedimentary rocks. Additionally, shale formations can act as both source and seal rocks (Walters, 2017), affecting the distribution and migration of hydrocarbons within a reservoir.

Thus, in order to create efficient methods for hydrocarbon production, such as optimizing well site, creating stimulation treatments, and putting enhanced oil recovery procedures in place, a full understanding of the qualities and traits of various rock types is essential (Deutsch & Hewett, 1996; Farzaneh & Sohrabi, 2013). Engineers and geoscientists can more accurately forecast the behavior of fluid flow by carefully researching the geological and petrophysical characteristics of reservoir rocks, which will also increase the productivity and profitability of oil and gas production.

Sandstone Reservoirs

Sandstone reservoirs are one of the most common types in the petroleum industry. They are composed of sand-sized grains that are held together by a cementing material, such as silica, calcium carbonate, or iron oxide. The porosity and permeability of sandstone reservoirs can vary widely depending on factors such as grain size, sorting, and cementation (Ajdukiewicz & Lander, 2010; Bo et al., 2021; Weimer & Tillman, 1982).

Carbonate Reservoirs

Another common form in the petroleum sector is carbonate reservoirs. Unlike sandstone, which are primarily composed of mineral grains, carbonate reservoirs are made up of calcite (CaCO_3) or dolomite ($\text{CaMg}(\text{CO}_3)_2$) minerals. These minerals can be formed in a variety of ways, including through the accumulation of marine organisms, chemical precipitation, or the alteration of existing rocks (Ahr, 2008; Z.-X. Xu et al., 2020). The properties of carbonate reservoirs, such as porosity and permeability, can be highly variable and are influenced by factors such as mineralogy, diagenesis, and fracturing (Kerans & Tinker, 1997).

In that order, dolomite and limestone, both primarily composed of calcium carbonate, are commonly encountered rock types in carbonate reservoirs. Dolomite reservoirs are commonly distinguished by their elevated levels of porosity and permeability, which frequently create advantageous circumstances for the extraction of oil and gas (Halley & Schmoker, 1983). It is noteworthy that the porosity and permeability of dolomite reservoirs are not uniformly high, owing to the influence of depositional environment, diagenetic history, and other geological factors (Spötl & Pitman, 1998; Warren, 2000). The porosity and permeability characteristics of limestone reservoirs can vary considerably due to various factors, including primary depositional textures, diagenesis, and subsequent fracturing (Hashim & Kaczmarek, 2019; Peacock & Mann, 2005; Spötl & Pitman, 1998).

Chalk, on the other hand, is a fine-grained, porous limestone composed mainly of the remains of tiny marine organisms. Chalk reservoirs are known for their low permeability and can present challenges for oil and gas production. However, fractures in chalk reservoirs can enhance their permeability and facilitate economic flow rates.

Brief Comparison

Sandstone and carbonate reservoir rocks differ fundamentally in two ways. Firstly, sandstones are formed from sediment that has been transported from one location to another (allochthonous), while carbonates are formed in their place of deposition (autochthonous). Secondly, carbonate minerals are more chemically reactive compared to sandstone minerals (Choquette & Pray, 1970; Moore, 2001).

Because of this contrast, large and systematic differences might be expected in their properties. Sandstone reservoirs tend to have larger pore spaces and more interconnected pore networks compared to carbonate reservoirs, making them generally more porous and permeable (Bjørlykke & Jahren, 2015).

However, a study (Ehrenberg & Nadeau, 2005) indicates that the occurrence of low-porosity siliciclastic reservoirs is relatively less frequent across all depths as compared to carbonates, which may be due to the greater prevalence of fractures in carbonate reservoirs. These fractures may play a significant role in enabling the flow of fluids and hence ensuring economic viability even in low-porosity carbonate rocks.

Additionally, in contrast to sandstone reservoirs that are usually composed of uniform interparticle pores and considered single-porosity systems, carbonate rock reservoirs are typically multiple-porosity systems that add to the petrophysical heterogeneity of the reservoirs (Mazzullo, 2004). Nonetheless, carbonate reservoirs can present challenges due to their heterogeneity and the potential for reservoir damage from acidizing treatments (Nadeau & Ehrenberg, 2006).

2.2.4. Fractures

Fractures are natural or induced features in rocks that can greatly affect fluid flow in reservoirs. Natural fractures are caused by geological processes that generate stresses and strains on rock formations, leading to the development of cracks and fractures. These fractures can create pathways for fluid flow and affect the porosity and permeability of the rock. They can also occur at different scales, from large faults that can extend for kilometers to tiny microfractures that are only visible under a microscope (Pyrak-Nolte et al., 1990; Schlumberger, 2023; Wu, 2015).

On the other hand, induced fractures, also known as hydraulic fractures or "fracking", are fractures that are created in the reservoir rock using hydraulic pressure. This is typically done by pumping fluid (usually water with additives) into the rock at high pressure, which creates fractures that propagate through the rock. These fractures can improve the permeability of the rock and increase the flow of oil or gas to the wellbore. Induced fractures are commonly used in unconventional reservoirs, such as shale and tight sandstone formations, where the natural permeability of the rock is low. However, there are also concerns about the environmental impacts of hydraulic fracturing, such as water contamination and induced seismicity (Khosravanian & Aadnoy, 2021; Kolawole & Ispas, 2020).

Fractured reservoirs (natural or induced) can have high permeability due to the presence of interconnected channels that allow fluids to flow more easily through the rock (Rueda et al., 2019). However, these fractures can also lead to unwanted water production during oil and gas operations. To mitigate this issue, water shutoff operations is often necessary to plug the fractures where water is likely to flow using gels or other materials that can reduce the permeability of the rock and prevent the movement of water (Zhang et al., 2020).

2.2.5. High Permeability Layers

High permeability layers are a common issue in many reservoirs, where certain layers of rock have a higher permeability than others (Tiab & Donaldson, 2016). This means that fluids, such as oil and water, are more likely to flow through these high permeability layers than through the surrounding rock. As a result, during oil and gas production, oil can become trapped in the less permeable layers, while water flows more easily through the high permeability layers and is produced along with the oil (Ruelland & Bu-Hindi, 2009). This can lead to increased water production, which can be costly and can reduce the overall efficiency of the well.

Conventional methods to mitigate the production of unwanted water include water shut off techniques, such as plugging the high permeability layers with gels or other materials (Sheng, 2010). Another approach is to use smart well completion techniques, which can help to control fluid production from different zones within the reservoir (Kerchner et al., 2000; Van Lopik et al., 2021). However, these methods can be challenging to implement and may not always be effective. New technologies, such as smart gels that can selectively target high permeability layers, are being developed to address this issue (Koochakzadeh et al., 2023). Overall, managing high permeability layers is a key challenge in the oil and gas industry, and effective solutions are critical to maximizing production and minimizing costs.

2.3. Laponite

Laponite is a synthetic, inorganic clay that has gained attention in the petroleum industry due to its unique properties as a gelling agent. It is a disc-shaped, platelet-like particle that has a diameter of approximately one nanometer and a thickness of around one nanometer (Liu et al., 2018; Willenbacher, 1996). Laponite is composed of magnesium, aluminum, silicon, and oxygen, and it has a negative surface charge that allows it to interact with cations in solution (Bonn et al., 1999).

Figure 2 depicts the structure of Laponite, with a chemical representation on the left and a nanocrystal geometry on the right. The chemical structure shows the arrangement of oxygen (red circles), silicon (green circles), hydrogen (white circles), and either magnesium or

lithium (purple circles). On the right side, the disk-like shape of the Laponite nanocrystal is visible, with a negatively charged surface and a positively charged edge.

When added to a solution, Laponite can form a three-dimensional network of particles, resulting in a gel-like substance that can be used to modify the rheological properties of fluids (Tomás et al., 2018).

Laponite has several advantages over other gelling agents, including its ability to rapidly gel at low concentrations, its compatibility with a wide range of fluids, and its stability over a wide range of temperatures and pH values (Au et al., 2015; Dawson & Oreffo, 2013; Ruzicka & Zaccarelli, 2011; Xiong et al., 2019). In the petroleum industry, Laponite gels have been used for various applications, such as water shut-off treatments (Aalaie & Youssefi, 2012; Tongwa et al., 2013), wellbore stabilization (Abdullah et al., 2022), and fracturing fluid design (Levine et al., 2009).

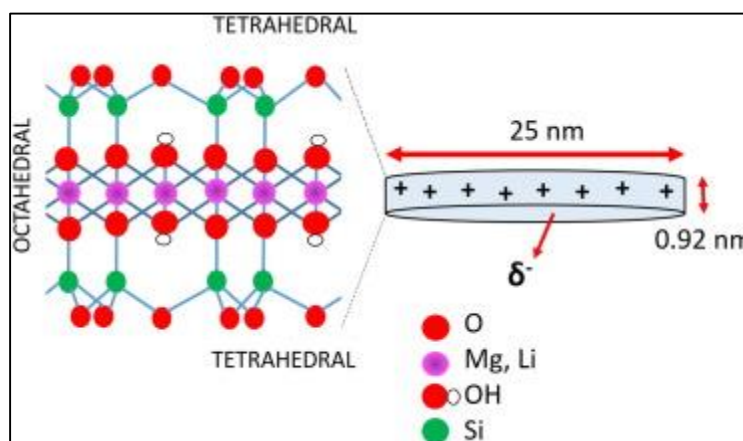


Figure 2. Schematic representation of the Laponite nanocrystal geometry (disk shape) and chemical structure. Taken from (Tomás et al., 2018).

2.3.1. Interaction with Ions

The physical properties of Laponite colloids are significantly influenced by the chemistry of the edge surface, owing to the small particle diameter of Laponite. When exposed to ionic solutes, Laponite particles tend to form gels through edge-face interactions. However, at elevated ionic strengths, they tend to flocculate. For instance, tetravalent pyrophosphate salts are commonly used commercially to prevent aggregation and enhance dispersion. This is achieved through complexation with the hydroxyl groups exposed at the particle edge, effectively screening them (Kim et al., 2020).

In this manner, cations and anions can interact with the interlayer space of Laponite clay, causing changes in the interlayer spacing and the interlayer electrostatic repulsion. These interactions can influence the hydration state of Laponite clay, consequently modifying the rheological properties of the suspensions.

2.4. HPAM

HPAM (Hydrolyzed Polyacrylamide) is a water-soluble polymer that is widely used in the oil and gas industry as a thickening agent for enhanced oil recovery (EOR) processes. HPAM is a synthetic polymer that is created by polymerizing acrylamide monomers in the presence

of a catalyst and a crosslinker. The resulting polymer has a high molecular weight and long chains that can entangle and thicken water-based fluids (Lewandowska, 2007).

In EOR applications, HPAM is typically used as a mobility control agent to reduce water production and increase oil recovery. When injected into the reservoir, HPAM can increase the viscosity of the injected water (Jung et al., 2013), which can help to sweep the oil towards the production wells. HPAM can also reduce the permeability of the reservoir to water (Fakher & Bai, 2018; Yadav et al., 2020), which can help to mitigate unwanted water production. However, HPAM can also interact with reservoir minerals and other components in the reservoir (Oliveira et al., 2019), which can lead to the formation of precipitates or gel-like structures that can reduce oil production if not managed properly.

2.5. Laponite – HPAM System

Laponite clay is a type of synthetic clay that has a unique structure consisting of plate-like particles. When Laponite clay is combined with a polymer, the clay particles become surrounded and dispersed in the polymer matrix (Tomás et al., 2018). The resulting material is a gel-like substance with properties that depend on the specific polymer and the type of Laponite clay used (Yang et al., 2022).

The formation of the gel is due to the interparticle interactions between the Laponite clay particles and the polymer (Tomás et al., 2018). The clay particles aggregate to form a network-like structure that gives the gel its viscoelastic properties. The polymer provides stability and enhances the mechanical strength of the gel (Suterio et al., 2022). This combination of clay particles and polymer results in a nanocomposite gel with mechanical, rheological, and chemical properties that make it useful for various applications, including water conformance in the oil and gas industry (Salehi et al., 2020; Tongwa & Bai, 2014). Figure 3 shows a representation of a nanocomposite formed by combining Laponite and polymer.

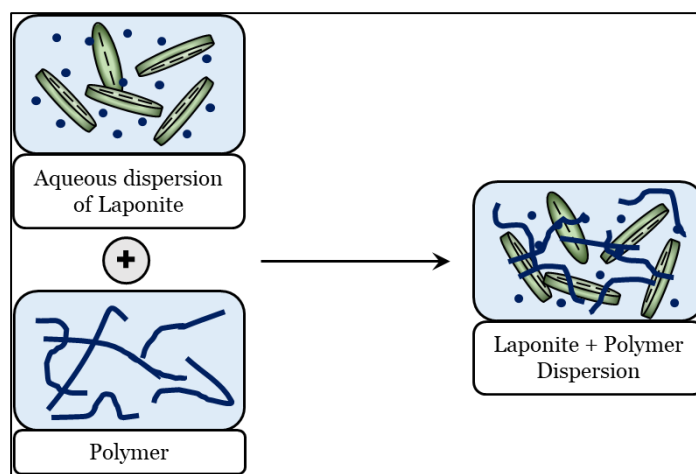


Figure 3. Schematic structure of nanocomposite formed by combining polymer and Laponite particles. Modified from (Fatnassi & Es-Souni, 2015).

That is the case when Laponite is combined with HPAM, the resulting material is a nanocomposite gel with unique properties that make it useful for water conformance applications in the oil and gas industry. HPAM is a water-soluble polymer that is commonly

used as a thickening agent due to its high molecular weight and ability to form strong and stable polymer chains in water (Li et al., 2014). When HPAM is combined with Laponite, it forms a hybrid system that combines the properties of both materials.

The addition of HPAM to the clay increases the gel's viscosity and mechanical strength, as well as its stability under high salinity and high temperature conditions (Aalaie & Youssefi, 2012). The Laponite particles act as a reinforcing agent, forming a network structure that provides additional mechanical support to the HPAM polymer chains. The resulting gel is also more resistant to shear and can maintain its structure for longer periods of time.

2.6. Gel Characterization

In the characterization of gels, various techniques are available, each offering valuable insights into different properties essential for their understanding and application. The aforementioned properties exhibit a diverse array of attributes, comprising of but not limited to, porosity, strength, permeability, and rheological behavior. Porosity assessments aid in the assessment of the empty spaces within the gel matrix, thereby offering valuable insights into its capacity to retain and convey fluids. Strength assessments are utilized to measure the mechanical characteristics of the gel and its ability to withstand deformation or breakage. The analysis of permeability facilitates comprehension of the gel's capacity to enable or impede the flow of fluid across its network. Moreover, investigating the rheological characteristics of gels enables us to gain insight into their flow attributes, such as stiffness, fluidity, and susceptibility to shear thinning.

This comprehensive characterization is crucial in elucidating the gel's performance and suitability for various applications. However, in our specific study, we focus on evaluating the behavior of gels over time, particularly their rigidity or viscosity. This aspect is of particular significance in fracture plugging, where the gel must maintain its structural integrity over an extended period, ensuring effective fracture sealing and long-term fluid control.

Nevertheless, lots of tests require special equipment or are invasive. For that reason, only some of the methods that are non-invasive, easy to use, simple, straightforward, and do not require special equipment, are listed below:

- Visual inspection: Simply observing the gel and checking if it has changed from a liquid-like to a solid-like appearance can be an effective way to determine the rigidity of a gel (Robert et al., 1987; Stavland et al., 2011).
- Penetration testing: Involves inserting a probe into the gel and measuring the resistance of the gel to penetration. As the gel becomes more rigid, it will provide more resistance to the probe, making it easier to determine when the gel has transitioned from a liquid to a solid (Hermansson, 1982; Jufri et al., 2018).
- Shear testing: Involves applying shear stress to the gel and measuring its response. As the gel becomes more rigid, it will provide more resistance to shear stress, making it easier to determine when the gel has transitioned from a liquid to a solid (Dokos et al., 2000).
- Compression testing: Involves applying compression stress to the gel and measuring its response. As the gel becomes more rigid, it will provide more resistance to

compression stress, making it easier to determine when the gel has transitioned from a liquid to a solid (Hermansson, 1982; Hernández et al., 1999).

- Dynamic Light Scattering (DLS): Shines a laser beam onto the gel and measures the scattered light patterns using a photodetector. The results are analyzed to determine the size and size distribution of the particles in the gel, which can provide valuable information about the gel's mechanical properties (Lang & Burchard, 1991; Shibayama & Norisuye, 2002).
- Gel Filtration Testing: In the standard procedure, puts a gel sample in a filtration apparatus, such as a Buchner funnel, and a vacuum is applied to draw the gel through the filter. The elapsed time for the gel to cease flowing through the filter is recorded and can be used to estimate the gel's rigidity (Raha et al., 2009). An alternative approach is to utilize a simpler filter that relies solely on gravity, which can still determine the rigidity of the gel.

3. Procedure and Materials

The present chapter provides a comprehensive account of the experimental procedures and materials employed in this research work. It is organized into four distinct sections, which are outlined as follows:

1. The first section presents the chemicals, materials, and equipment employed in this research.
2. The following section delineates the methodology for performing bulk tests, which include tests for gelation time, sensitivity to cations and sensitivity to temperature.
3. The candidates exhibiting the most potential, as determined by the bulk tests, were subjected to a filtration test to evaluate the viability of their injection.
4. The final section provides a detailed account of the core flooding tests, including the setup employed and the procedure and parameters implemented.

The experimental procedures implemented in this research are illustrated in Figure 4 through a schematic diagram.

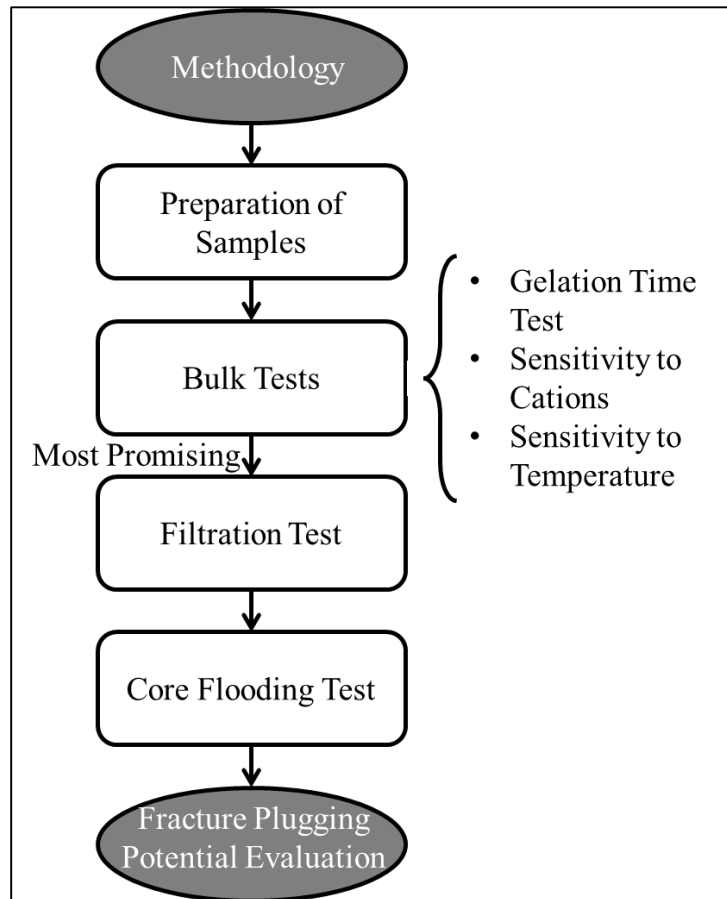


Figure 4. Schematic diagram of the methodology

3.1. Chemicals, Materials and Equipment

3.1.1. Chemicals

The chemicals used in this study included Laponite and HPAM. Additionally, for cation sensitivity tests, two different salts were employed: potassium chloride and calcium chloride.

Initially, two types of Laponite were examined: Laponite RD and Laponite S482, both acquired from the supplier BYK Additives Inc. Laponite RD is known for its applications as a gelling agent, as it is capable of forming a gel even at low clay concentrations and room temperature. It is composed of Silicic acid, lithium magnesium sodium salt.

Laponite S482, on the other hand, is a grade that forms stable aqueous solutions and is typically used for protective films rather than rheology applications. However, it was chosen in this study due to its superior stability compared to other Laponite grades and its ability to form gels when the temperature is increased. It primarily consists of Laponite RD, with a composition containing approximately 3 to 10% (1-hydroxyethylidene) bisphosphonic acid, sodium salt.

The HPAM polymer used, Flopaam 3630 S, was sourced from the supplier SNF S.A.S. It possesses a molecular weight of 18,000,000 daltons and was selected due to its widespread use as a gelling agent.

3.1.2. Materials and Equipment

In the bulk tests, the following equipment was utilized:

- Test tubes/conical flasks: These were used for storing the samples during the experiments.
- Weighting balance: A Mettler Toledo PB303 balance was employed to measure the chemicals and solutions accurately.
- Magnetic stirrer: The Heidolph MR hei-standard stirrer was utilized to mix the solutions effectively.
- Vortex shaker: A vortex shaker was employed to mix the gel solutions in the test tubes.
- Oven: Samples were heated at 50 or 70°C using an oven.

3.2. Bulk Test

The bulk tests conducted in this study involved the chemical screening and characterization of different gel samples made from Laponite and HPAM. Various methodologies exist for the preparation of nanocomposites, and in this case, a simple approach for gels intended for water control treatment was implemented, as described by (Mohammadi et al., 2015). However, it is important to note that the use of crosslinkers was not within the scope of this project, which distinguishes it from the aforementioned study.

The bulk tests were divided into three parts: gelation time tests, sensitivity to cation tests, and sensitivity to temperature tests. It should be clarified that these tests were occasionally conducted simultaneously using different samples. Additionally, the cation sensitivity and temperature sensitivity analyses were performed both separately and in combination. It is

worth mentioning that the interaction in deionized water was primarily evaluated, but the study was also extended to investigate the interaction in seawater.

3.2.1. Sample Preparation

The bulk test samples were prepared in 10 mL sample tubes. The compositions varied, with the Laponite concentration ranging from 0.5% to 4.0% by mass, equivalent to 0.05 g and 0.40 g, respectively, in a sample. The polymer concentration ranged from 0.00% to 0.30%, equivalent to a sample without HPAM and one with 0.03 g, to make up a total mass of 10 g using deionized water.

Based on information from the supplier, it was known that a sample with 4.0% of Laponite RD was capable of gelling (BYK, 2014). Hence, this concentration was selected for further experimentation. Additionally, samples were prepared with 0.5%, 1.0%, and 1.5% of this grade of Laponite, and their interactions were analyzed with 0.00%, 0.15%, and 0.30% HPAM for each clay concentration.

The steps for sample preparation are outlined as follows:

1. Laponite dispersion:

The appropriate amounts of Laponite and deionized water were measured into the test tubes (without grading). The clay and water were mixed using a vortex mixer set at speed 7 for approximately 1 minute or until a clear and homogeneous solution was obtained. If homogeneity was not achieved, a magnetic stirrer was used until a clear and homogeneous solution was obtained. The test tubes were closed and stored at room temperature

2. HPAM stock:

To prepare a 1.0 w% HPAM bulk solution, 1.00g of HPAM was added to a 100 mL measuring flask, and it was filled with deionized water up to the 100 mL mark. The sample was stirred on a magnetic stirrer for 2 hours until a uniform solution was formed. The solution was stored in the closed measuring flask at room temperature for future use.

3. Nanocomposite gel preparation:

The required amounts of Laponite and deionized water were measured into the test tubes (without grading) according to the specifications. The clay and water were mixed using a vortex mixer set at speed 7 for approximately 1 minute or until a clear and homogeneous solution was obtained. If homogeneity was not achieved, a magnetic stirrer was used until a clear and homogeneous solution was obtained. The polymer solution was added to the test tube. The solution was mixed using a vortex mixer set at speed 7 for approximately 1 minute or until a clear and homogeneous solution was obtained. The tubes were kept closed at room conditions.

The same procedure was performed for both Laponite RD and Laponite S482. Subsequently, the interaction of the nanocomposites with seawater, instead of de-ionized water, was studied. For this purpose, samples with the same concentrations of Laponite S482 and 0.15% HPAM were prepared using seawater.

Table 2. Composition of Synthetic Seawater.

Salt	Chemical Formula	Concentration of salt in SSW (g/l)	Supplier
Sodium Chloride	NaCl	23,38	Merck Chemicals
Sodium Sulfate	Na ₂ SO ₄	3,42	Merck Chemicals
Sodium Carbonate	NaHCO ₃	0,17	Merck Chemicals
Potassium Chloride	KCl	0,75	Merck Chemicals
Magnesium Chloride Hexahydrate	MgCl ₂ *6H ₂ O	9,05	Sigma-Aldrich
Calcium Chloride Dihydrate	CaCl ₂ *2H ₂ O	1,91	VWR international

The composition of synthetic seawater utilized in the preliminary testing of polymer and clay gelation interactions is presented in Table 2. The seawater solution described was produced by introducing the salts in their designated quantities into one liter of deionized water, followed by agitation of the solution using a magnetic stirrer for a duration of 24 hours.

3.2.2. Gelation Time Test

After the preparation of the samples, the gelation time was tested by visually inspecting the gels. The test tubes were gently inverted, and the properties of the gel were evaluated at room temperature using gel codes based on the classification by (Skrettingland et al., 2012). The initial inspection, conducted immediately after the sample preparation, was considered as time 0. Subsequent inspections were performed at 2, 4, 6, 24, and 28 hours.

Table 3. Gel Classification Code (Ogienagbon, 2019)

Gel Code	Gelant status upon gentle shakes/inversion of tubes
1	Seems to have original viscosity (no gel)
2	Some increase in viscosity (freely flowing gel)
3	Highly viscous and deformable flowing fluid
4	Deformable upper part with high flow resistance
5	Rigid gel (no flow or deformation)

This classification did not provide a precise quantitative description of hardness or viscosity since no specific measurement instrument was used. It is important to note that there were significant gaps between the values, making it challenging at times to classify between one code and another. However, despite these limitations, the classification system offered a

quick, simple, and reliable way to assess the gelation process. It allowed for a visual observation of the increase in sample rigidity and identified samples that failed to form a gel.

3.2.3. Sensitivity to Temperature

After the gelation time test, the temperature sensitivity of each sample was evaluated. To simulate reservoir conditions, the prepared samples were placed in an oven set at 50 °C. This step aimed to observe the effects of temperature increase on the gelants, considering that reservoir temperatures are typically higher than ambient temperatures. By subjecting the samples to elevated temperatures, we could assess any changes or behaviors exhibited by the gelants under such conditions.

To monitor their gelation progress, the samples were periodically evaluated at specific time intervals of 2, 4, 6, and 24 hours. During each evaluation, the samples were removed from the oven, their gel codes were assessed according to the classification provided in Table 3, and then they were promptly returned to the oven to maintain the temperature of 50 °C for subsequent evaluations.

The same procedure was done for studying the sensitivity at 70 °C. This temperature is more similar to the common reservoir temperatures in the North Sea. Afterwards, 70 °C was preferable for the latest experiments.

3.2.4. Sensitivity to Cations

To assess the sensitivity of the samples to cations, brines containing KCl and CaCl₂ were added to Laponite S482 samples, both with and without polymer. The salt solutions were prepared by measuring the desired amount of salt (KCl or CaCl₂) in a graduated flask, followed by adding water to reach the desired volume. The solutions were then mixed using a magnetic stirrer for 24 hours to ensure homogeneity.

For the Laponite-salt or nanocomposite-salt samples, the following procedure was followed: First, the desired amount of Laponite was added to the test tube. De-ionized water was then added and mixed as described in section 3.2.1. Sample Preparation, until a uniform mixture was obtained. It should be noted that part of the water was reserved for the addition of brine and polymer solution. Next, the salt solution was added to the test tube and mixed using a vortex mixer at speed 7 until homogeneity was achieved. Finally, the polymer solution was added and mixed in the vortex. If uniformity was not achieved, a magnetic stirrer was used until all the solution was dissolved in the sample.

For these samples, gelation time tests were conducted at both room temperature and 70°C to evaluate the gelation properties.

3.2.5. Chemical Screening

After evaluating the sensitivity of Laponite to temperature and cations in the previous sections, a screening process was conducted to select the two most promising gel systems for further investigation. The objective was to identify gelant formulations that exhibited favorable properties in terms of gelation behavior and stability.

The screening process involved variations in the concentrations of Laponite S482, hydrolyzed polyacrylamide (HPAM), and calcium chloride (CaCl₂). Two systems were specifically targeted for evaluation: the Laponite S482-CaCl₂ system and the nanocomposite

system with the addition of HPAM. This selection allowed for a comparison between a system comprising solely Laponite and a nanocomposite system incorporating this clay.

For the screening of the Laponite S482-CaCl₂ system, different proportions of Laponite S482 were combined with CaCl₂, aiming to determine the minimum concentration required to form a gel. The objective was to identify the composition that exhibited the most favorable gelation characteristics.

Regarding the nanocomposite system, the concentration of Laponite S482 was fixed at 2.0% based on the results obtained from the previous screening. This concentration was selected to maintain consistency and facilitate comparisons. Two variations of HPAM, 0.15% and 0.30%, which had been studied in previous bulk experiments, were incorporated into the nanocomposite system. Various concentrations of CaCl₂ were then evaluated to achieve the formation of a rigid gel.

By conducting this chemical screening, the aim was to identify the most promising gel systems that demonstrated desirable gelation behavior and potential for fracture plugging. The selected systems were further evaluated in subsequent experiments to assess their performance and effectiveness.

3.3. Filtration Test

In order to evaluate the injectivity of the gelants, a filtration test was conducted on the most promising samples selected during the chemical screening. A filter TMTP04700 (hydrophilic polycarbonate membrane filter with a pore size of 5.0 μm) supplied by Merck Millipore Ltd., was utilized for this purpose.

The setup for the filtration test is illustrated in Figure 5. The fluid being tested is contained in a piston cell, and nitrogen is injected at one side of the piston at a pressure of 2 bar. The fluid produced during the test is collected in a beaker placed on a scale, which enables continuous monitoring of the weight. A time-weight graph is constructed for the purpose of examining the correlation between time and the mass of the produced fluid.

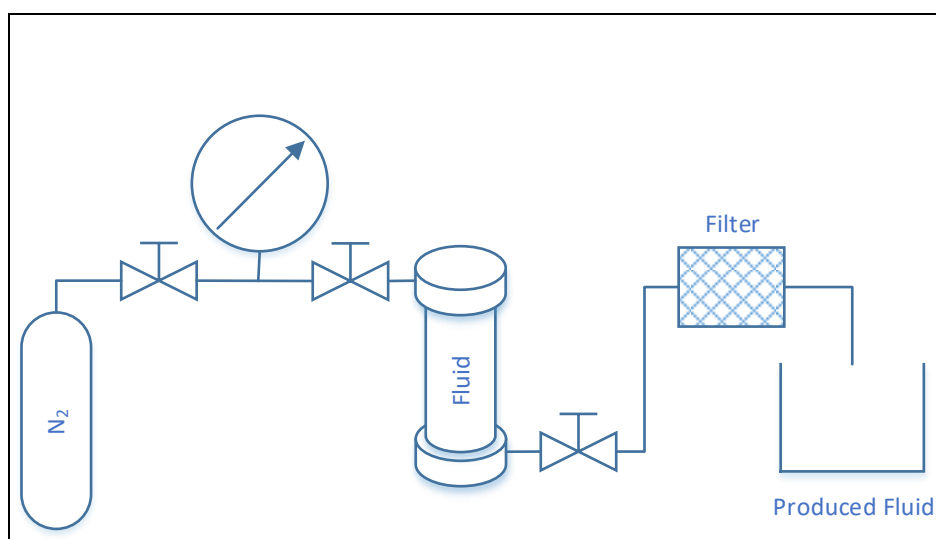


Figure 5. Schematic setup of filtration test.

3.4. Core Flooding Test

The core flooding tests were conducted to evaluate the potential of the gels in plugging fractures in chalk. Figure 6 illustrates the schematic setup used for these experiments. During the test, either the gelant or distilled water is injected into the core. The second piston cell contains oil and was used to maintain confining pressure. The pressure transducer measures the differential pressure (dP), and a back pressure regulator is set at 10 bar.

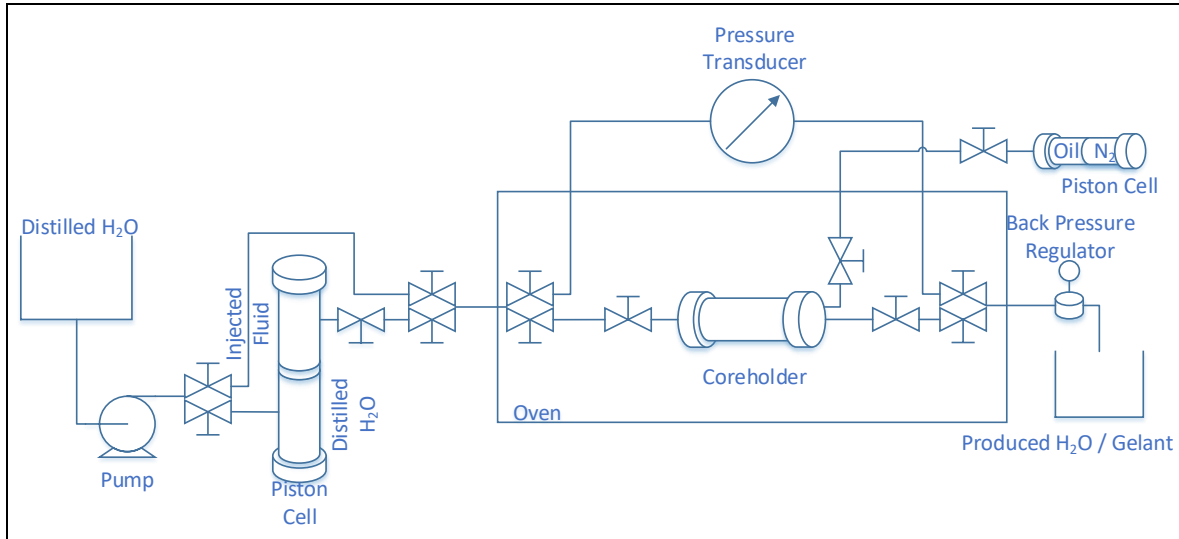


Figure 6. Schematic setup of core flooding test.

Two systems, selected as the promising ones in the chemical screening and then tested to secure gel placement in the filtration test, were made, first with 2.0% of Laponite S482 and 0.20% CaCl₂, and the second with 2.0% Laponite S482, 0.15% HPAM and 0.15% CaCl₂. The gelant solutions were prepared as follows:

1. Measure the desired quantity of deionized water in a flask.
2. Add the desired quantity of Laponite to the flask and stir at 400 rpm to ensure uniform dispersion.
3. Add the desired quantity of CaCl₂ to the solution from step 2.
4. Stir the solution for at least 40 minutes at 700 rpm.
5. In another flask, measure the desired quantity of deionized water.
6. Add the desired quantity of HPAM to the flask and mix with a magnetic stirrer.
7. Measure the desired quantity of HPAM solution and add it to the solution prepared in step 3.
8. Mix the combined solution with a magnetic stirrer.

The preparation of the core sample involves measuring its dimensions and mounting it into the core holder. Vacuuming is performed to remove excess air, and the weight of the core holder is measured. The core is then filled with deionized water, and its weight is determined. The pore volume is calculated by subtracting the weight of the dry core from the weight of the saturated core and dividing it by the water density. The porosity is calculated using the next equation

$$\Phi = \frac{\text{Pore Volume}}{\text{Bulk Volume}}$$

To determine the absolute permeability of the core, deionized water is injected at room temperature using flow rates of 0.1, 0.2, and 0.3 ml/min until the dP stabilizes. The absolute water permeability is calculated by determining the slope of the pressure gradient versus the flow rate using Darcy's equation.

To create a fracture model in the core sample, the core is demounted, and a fracture with a diameter of 4 mm is created throughout the length of the core. The fracture is then packed with glass beads within the size range of 43-52 μm .

After creating the fracture model, the core sample is remounted, and a 3 mm thick slice of Bentheimer core with a permeability of approximately 2000 md is used as a filter at the inlet and outlet of the core to prevent the production of glass beads. Deionized water is injected to measure the permeability using flow rates of 1, 2, and 3 ml/min.

For gel injection and activation, the oven is turned on and set to 70°C. Once the temperature stabilizes, the prepared gelant is injected into the core at a flow rate of 0.5 ml/min until the gelant is produced. The core is left for 2 days to allow sufficient time for gel formation. This step accounts for the fluid interacting with the chalk, even though the gelants typically form a gel in less than 24 hours. This approach mimics field conditions where chemicals are injected from the surface at room temperature to the reservoir at high temperature.

After gel injection and activation, the oven is turned off, and the core is left to cool to room temperature. Deionized water is injected at various flow rates until the dP stabilizes. The corresponding dP is recorded for each flow rate, and the core's permeability to water is calculated using Darcy's equation by determining the slope of the pressure gradient versus the flow rate.

4. Results and Discussion

This chapter presents the results and analysis of the laboratory tests conducted as part of this thesis. It is divided into three sections: Bulk test, where various gelling agents were screened to identify the most suitable candidates; Filtration test, which evaluated the injectability of the selected candidates for fracture packing; and Core flooding test, which assessed the potential of the most promising gelants to effectively plug chalk fractures.

Each section focuses on specific aspects of the gelants' performance and suitability for fracture plugging. The Bulk test aimed to identify the gelants that exhibited the desired gelling properties, particularly, gel formation on specific conditions. The Filtration test further evaluated the injectability of the most promising gelants, considering factors like pressure and filtration efficiency. Lastly, the Core flooding test assessed the ability of the selected gelants to effectively plug chalk fractures, considering parameters such as permeability reduction and the Residual Resistance Factor.

By analyzing the results obtained from these tests, valuable insights can be gained regarding the performance and potential application of the gelants in the context of fracture plugging. The findings from this chapter contribute to a comprehensive understanding of the suitability and effectiveness of the tested gelants, paving the way for further development and potential implementation in real-world field scenarios.

4.1. Bulk Tests

This section presents the results of the bulk tests and chemical screening conducted to identify the most promising candidates for further evaluation in subsequent tests. The bulk tests aimed to screen various gelling agents and assess their performance in terms of gelling properties, such as gel strength, stability, and viscosity, through a gel code described in table 3, where 1 indicates the lowest viscosity and 5 is a rigid gel.

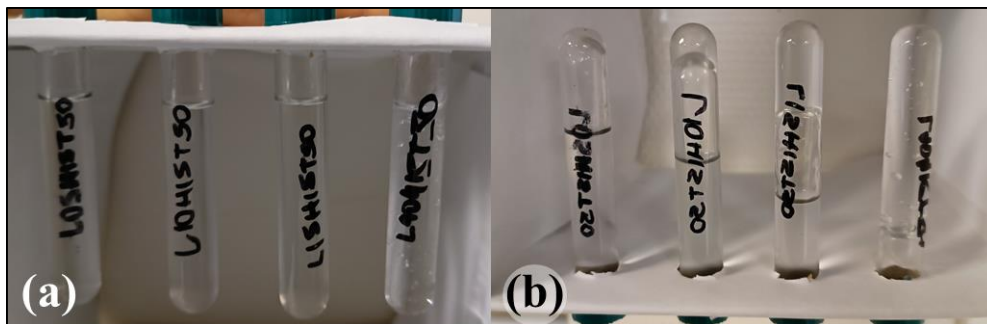


Figure 7. Example of samples during bulk test. Before (a) and after (b) turning upside down.

Figure 7 presents an illustrative example of the bulk test. The left image displays the samples in a resting state, while the right photo showcases the samples after being inverted. The significant variation in viscosities among these samples is evident, enabling the evaluation of their gel codes. In that photo, the leftmost sample exhibits a gel code of 1 or 2, as it quickly flows down without resistance, indicating a low viscosity comparable to that of deionized water at ambient conditions. Conversely, the subsequent sample demonstrates slower flow,

while the third sample displays partial gelation. The rightmost sample, on the other hand, exhibits complete gelation, remaining stationary when turning upside down.

The chemical screening process involved testing different combinations and concentrations of gelling agents, along with other additives if applicable, to determine their compatibility and potential synergistic effects. This screening process helped narrow down the candidates and identify the formulations that exhibited the desired gelling characteristics.

4.1.1. Characterization of Laponite RD Gels in Deionized Water

The initial experiments focused on investigating the gelation behavior of Laponite RD by varying the clay concentration. Bulk tests were conducted to assess the outcomes of these experiments, and the results are depicted in Figure 8.

As depicted in the figure, a concentration of 4.0% w/w Laponite RD is necessary to achieve a rigid gel at room temperature. Samples with lower clay concentrations did not exhibit any change in viscosity; instead, they remained as stable liquid solutions. This outcome can be attributed to the insufficient concentration of Laponite, which prevents the clay discs from forming a cohesive structure. Consequently, the Laponite remains dissolved in the water without undergoing significant structural changes.

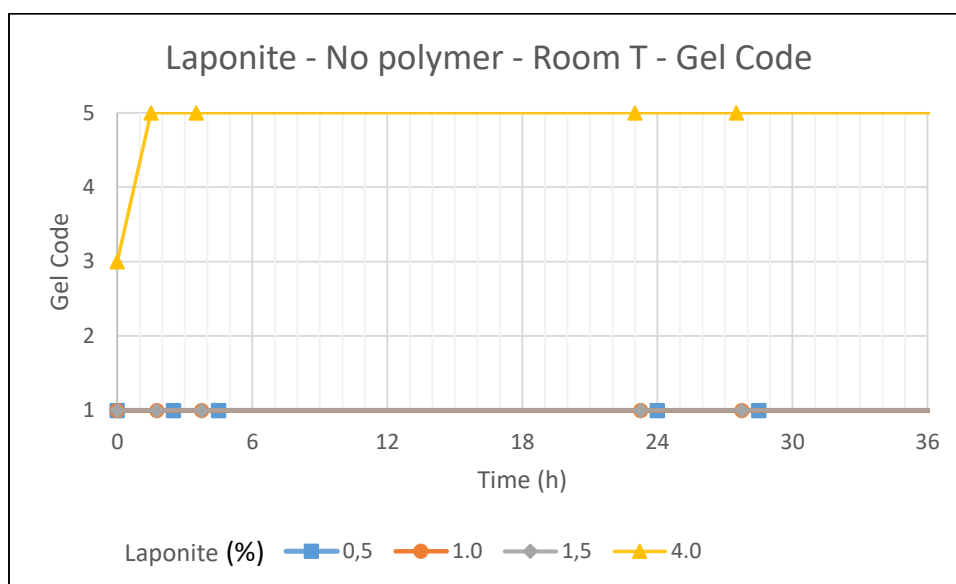


Figure 8. Gel code for gelants varying Laponite concentration at room temperature. Note that 0.5, 1.0 and 1.5 % Laponite lines are overlapping.

Subsequently, bulk tests were conducted with the addition of polymer. Two concentrations of HPAM (0.15% and 0.30% w/w) were investigated, while the same concentrations of Laponite RD were utilized for both polymer concentrations. Figure 9 illustrates the results of these tests for various Laponite concentrations in combination with 0.15% HPAM, and figure 10 shows the results for 0.30% HPAM.

For the higher concentration of Laponite, which already formed a gel in the absence of polymer, the addition of 0.15% HPAM did not result in any significant changes. The gel retained its rigid structure. On the other hand, for Laponite concentrations below 1.5%, rigid gel formation was not observed, but there was an increase in viscosity to gel code 2. This

increase could potentially be attributed to the presence of HPAM, as it is known to exhibit crosslinking effects with Laponite. This interaction between the polymer and Laponite is likely the primary cause of the slight viscosity enhancement observed.

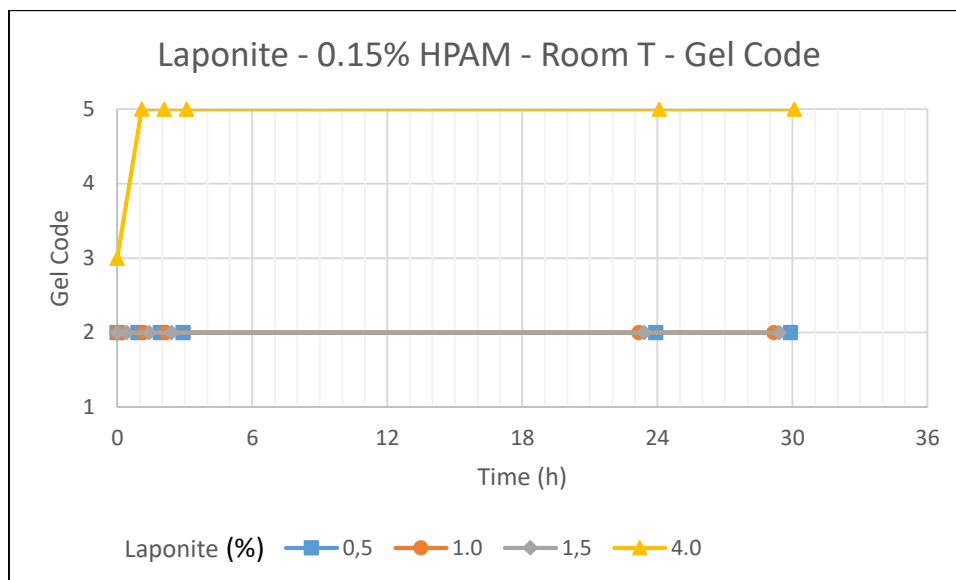


Figure 9. Gel code for nanocomposites with 0.15 % HPAM and varying Laponite concentration at room temperature. Note that 0.5, 1.0 and 1.5 % Laponite lines are overlapping.

When a higher amount of HPAM (up to 0.30%) was added, the mixture for the nanocomposite containing 4.0% Laponite encountered difficulties, as gel formation began during the preparation process, even with agitation. However, for the other samples, better results were achieved with this increased polymer concentration.

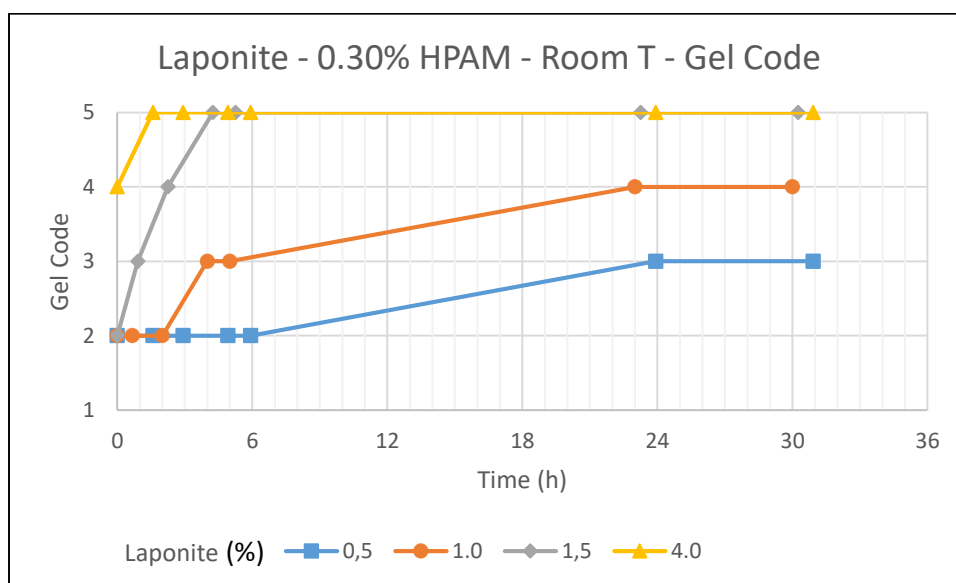


Figure 10. Gel code for nanocomposites with 0.30 % HPAM and varying Laponite concentration at room temperature.

For the combination of 1.5% Laponite RD and 0.30% HPAM, gel formation occurred at room temperature and occurred within the 4 hours after complete mixing. The gel remained stable for at least two weeks, indicating its long-lasting properties. This can be attributed to the crosslinking effect, where the polymer chains interact with the Laponite discs, leading to the formation of larger structures. As a result, the viscosity increases and eventually a gel is formed.

The impact of the higher HPAM concentration is also evident in the samples with lower Laponite concentrations. For instance, with 1.0% Laponite, there is a significant enhancement in sample rigidity to gel code 4, and a partial gel is formed. However, the gel slowly begins to flow after a few seconds. In the case of 0.5% Laponite, the viscosity increases to gel code 3, but no gel formation is observed.

This indicates that the incorporation of HPAM successfully enhances the rigidity of the gelant derived from Laponite and can ultimately lead to gel formation when both the Laponite and polymer concentrations are adequate. However, it is important to note that excessively high concentrations of either component can result in premature gelation, which may hinder the production of the desired nanocomposite. Therefore, finding the optimal balance between the concentrations of Laponite and HPAM is crucial to achieve the desired rheological properties without encountering issues related to early gelation.

4.1.2. Sensitivity to Temperature of Laponite RD Gels

To investigate the sensitivity of the gelants, samples with the same concentrations were subjected to an oven at 50 °C, considering that hydrocarbon reservoirs often have elevated temperatures. The results of these sensitivity tests are presented in the following figures.

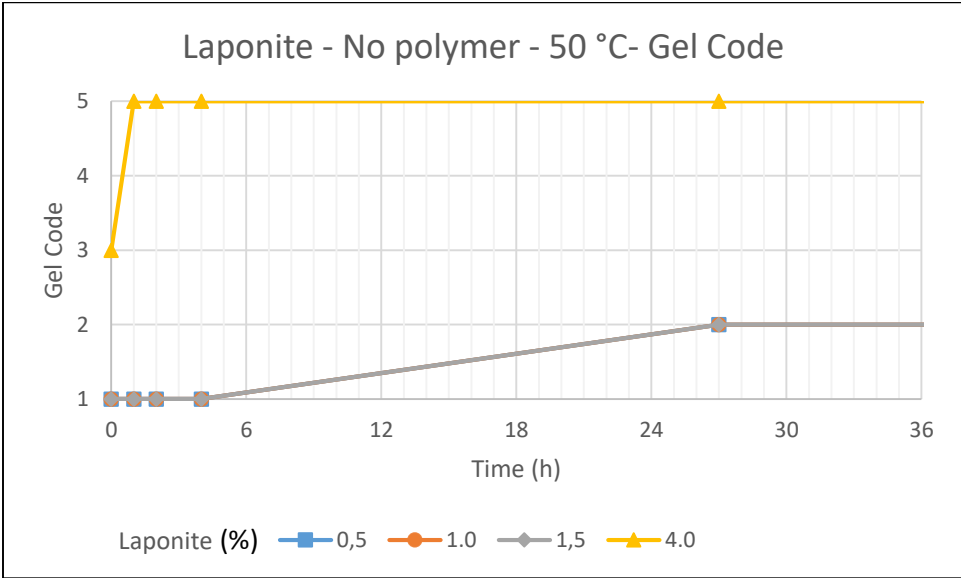


Figure 11. Gel code for gelants varying Laponite concentration at 50 °C. Note that 0.5, 1.0 and 1.5 % Laponite lines are overlapping.

Figure 11 displays the behavior of samples containing only Laponite RD. Minor changes were observed in samples with lower clay concentrations. After approximately 24 hours, a

slight increase in viscosity was noted. Although minimal, this observation is significant as it indicates that temperature can have a positive impact on the final stiffness of the gelant. This finding deviates from the supplier's indication that an increase in temperature would solely accelerate the gel formation (BYK, 2014), as demonstrated by the sample with 4.0% Laponite RD.

Figure 12 illustrates the temperature sensitivity of samples containing 0.15% HPAM. At 50 °C, two samples exhibited notable changes. The sample with 1.0% Laponite RD and 0.15% HPAM experienced an increase in viscosity to gel code 3, although insufficient to form a gel, while the sample with 1.5% Laponite and 0.15% HPAM partially formed a gel, thus it got gel code 4. This suggests that temperature influences the crosslinking effect, resulting in the formation of more stable structures.

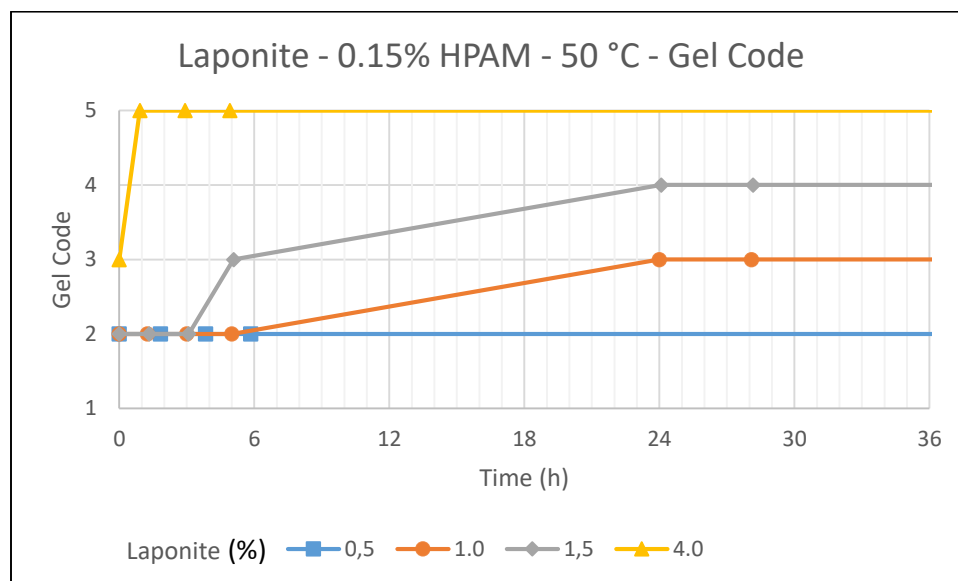


Figure 12. Gel code for nanocomposites with 0.15 % HPAM and varying Laponite concentration at 50 °C.

Figure 13 presents the results for nanocomposites with 0.30% HPAM. It is important to note that the sample with 4.0% Laponite RD was excluded from this study due to its pre-existing gel formation and challenging mixing process. However, all other samples exhibited a similar pattern, with reduced time required to reach their final state. For instance, the nanocomposite with 1.5% Laponite RD and 0.30% HPAM formed a gel in less than 2 hours compared to the 4-hour duration at room temperature. Lower concentrations reached their final state in approximately 4 hours, indicating no substantial change or stable gel formation compared to the room temperature test.

Overall, the sensitivity tests demonstrate that temperature can influence the rheological behavior of the gelants, leading to accelerated gel formation or increased stiffness. These findings are crucial for understanding the performance and potential application of the gel systems in reservoir conditions.

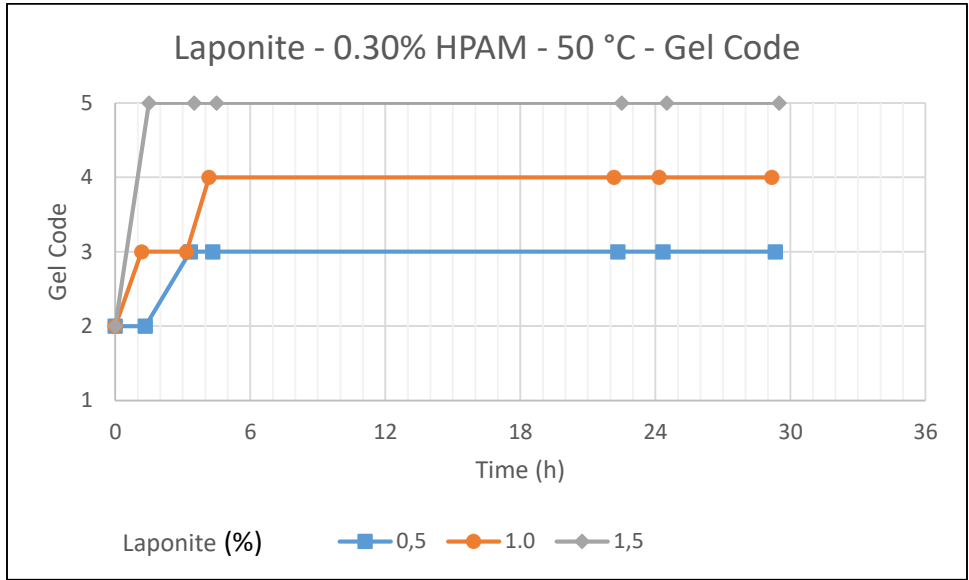


Figure 13. Gel code for nanocomposites with 0.30 % HPAM and varying Laponite concentration at 50 °C.

4.1.3. Characterization of Laponite S482 Gels in Deionized Water

Considering the supplier's claim of higher stability of Laponite S482 in liquid solution at room temperature (BYK, 2014) and its potential for gel formation at elevated temperatures, the focus shifted to studying this type of Laponite. Similar to the previous experiments with Laponite RD, the behavior of Laponite S482 was investigated with different concentrations of both Laponite and HPAM, maintaining the same concentrations as in the previous tests.

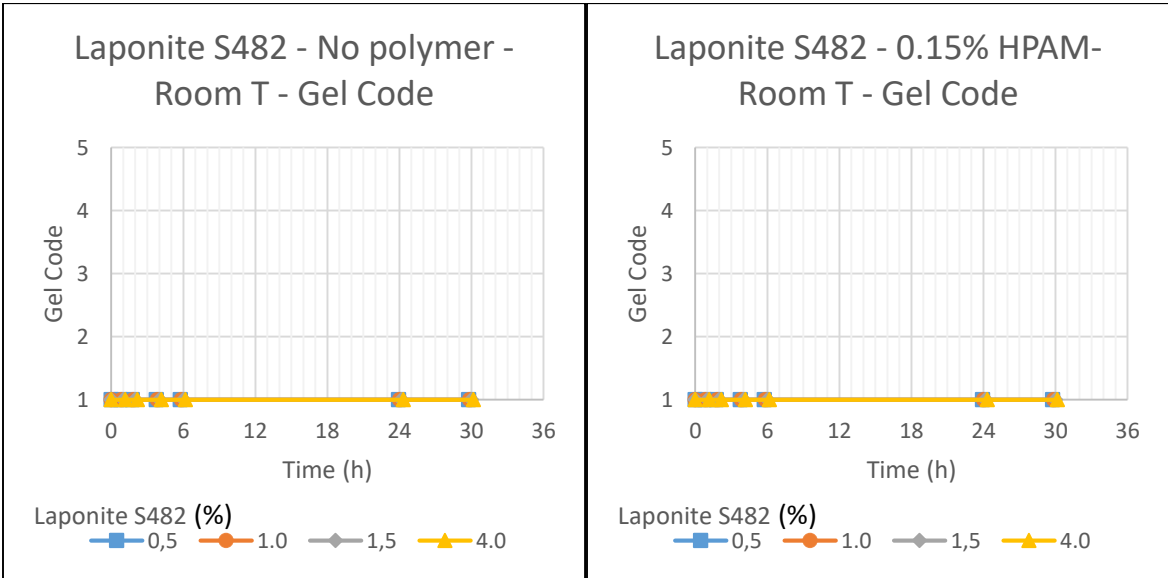


Figure 14. Gel code for gelant systems at room temperature with different Laponite S482 concentration (left graph), and same concentrations of Laponite S482 with 0.15 % HPAM (right graph). Note that 0.5, 1.0, 1.5 and 4.0 % Laponite S482 lines are overlapping in both plots.

Figure 14 displays the results obtained from these experiments, clearly indicating that Laponite S482 solution remain stable as a liquid solution at room temperature. Specifically, up to a concentration of 4.0% Laponite S482 and 0.15% HPAM, no gel formation was observed, suggesting that the clay remains in a dispersed state without significant structural changes.

4.1.4. Sensitivity to Temperature of Laponite S482 Gels

In parallel to the previous type of Laponite (RD), the influence of temperature on the gelants derived from Laponite S482 was also examined. Figure 15 illustrates the outcomes obtained at 50 °C, while Figure 16 displays the results at 70 °C. This temperature range was chosen to simulate the conditions commonly encountered in reservoirs, where temperatures often exceed 70 °C.

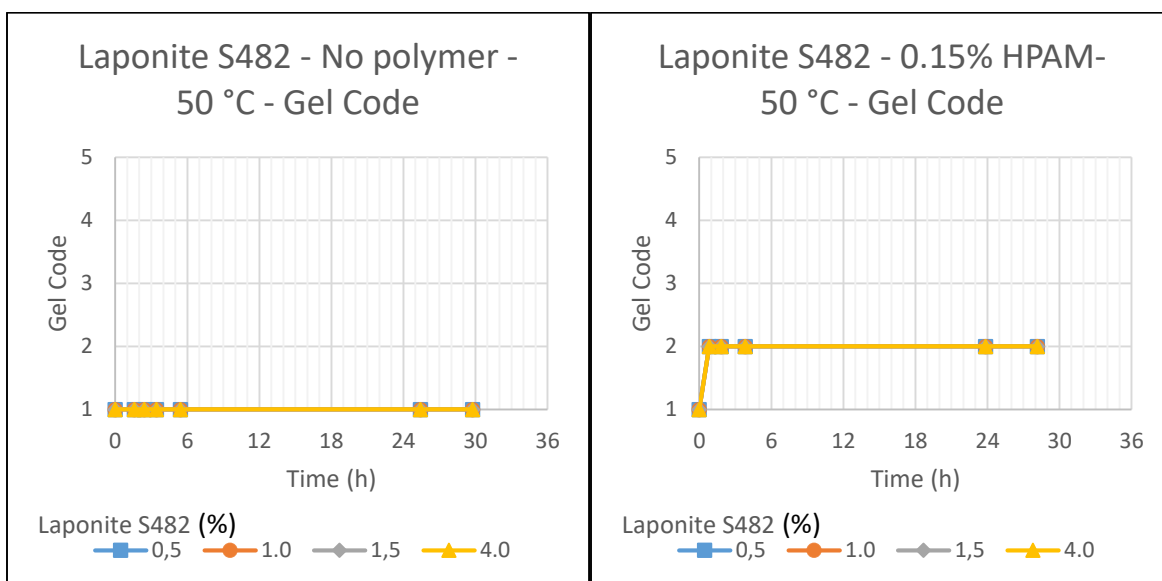


Figure 15. Gel code for gelant systems at 50 °C with different Laponite S482 concentration (left graph), and same concentrations of Laponite S482 with 0.15 % HPAM (right graph).

Note that 0.5, 1.0, 1.5 and 4.0 % Laponite S482 lines are overlapping in both plots.

The observations made at both temperatures were consistent. In the case of pure Laponite S482, there were no notable changes in the gelant as the temperature increased. The solution remained a stable liquid with consistent viscosity throughout the test. However, when 0.15% HPAM was added to the mixture, a slight increase in viscosity was observed with rising temperature. Despite this initial change, the samples maintained their stability and did not progress towards gelation.

The gelants containing 0.30% HPAM displayed similar behavior, aligning with the observations made for the lower concentration of the polymer. Although the specific graphs are not depicted here, the results were essentially identical to those obtained with the lower HPAM concentration.

These findings indicate that Laponite S482, in combination with HPAM, exhibits stability in liquid form at both ambient and elevated temperatures, without significant gelation occurring within the tested temperature range up to 70 °C.

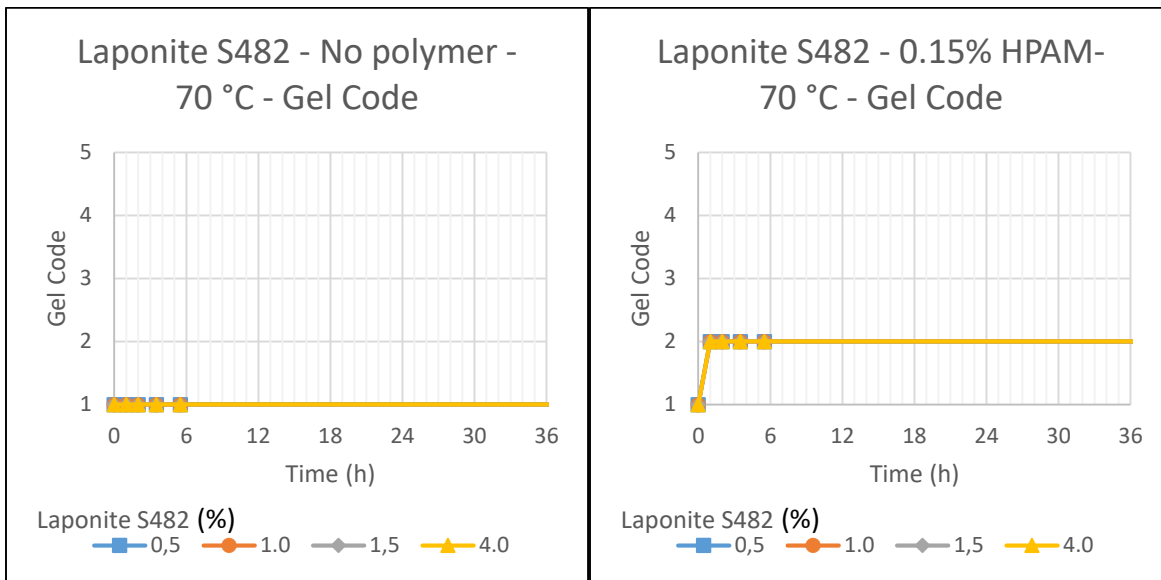


Figure 16. Gel code for gelant systems at 70 °C with different Laponite S482 concentration (left graph), and same concentrations of Laponite S482 with 0.15 % HPAM (right graph). Note that 0.5, 1.0, 1.5 and 4.0 % Laponite S482 lines are overlapping in both plots.

4.1.5. Characterization of Laponite S482 Gels in Seawater

This section presents the results obtained for the samples prepared with synthetic seawater, as a substitution for deionized water, across all the concentrations studied in Section 4.1.3. The sensitivity to temperature was also analyzed.

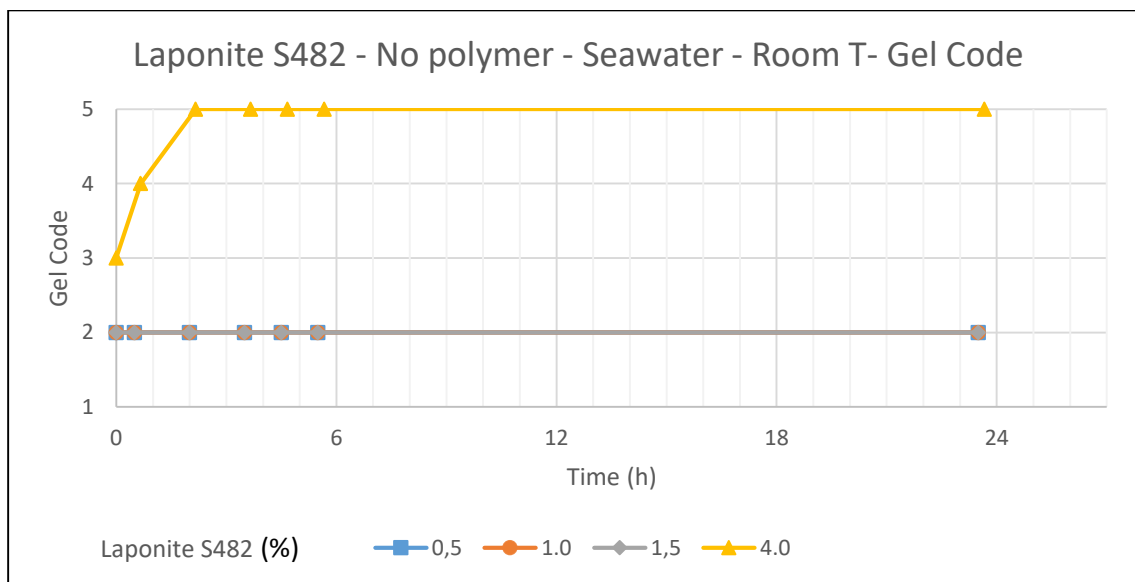


Figure 17. Gel code for gelants with different Laponite S482 concentration in seawater at room temperature. Note that 0.5, 1.0 and 1.5 % Laponite S482 lines are overlapping.

Figure 17 illustrates the outcomes for the samples containing only Laponite S482 at room temperature. The lower concentrations of Laponite exhibited a slight increase in viscosity, while the sample with 4.0% Laponite underwent a significant transformation, forming a gel

in approximately 2 hours. It is worth noting that a discernible rise in viscosity was observed for this particular sample, even in the midst of the agitation process. These distinct results can be attributed to the presence of ions in seawater, which facilitate the formation of structures by interacting with the surface and edges of the Laponite disks.

The effect of elevating the temperature to 50 °C is depicted in Figure 18. As expected, the temperature primarily influenced the time required to reach the final state. Lower concentrations of Laponite did not experience notable changes with increased temperature, while the sample with 4.0% Laponite S482 gelled more rapidly. It is important to note that this particular sample already exhibited relatively quick gelation at room temperature. Hence, while the temperature increase had some effect, it did not cause a significant alteration in the overall outcome.

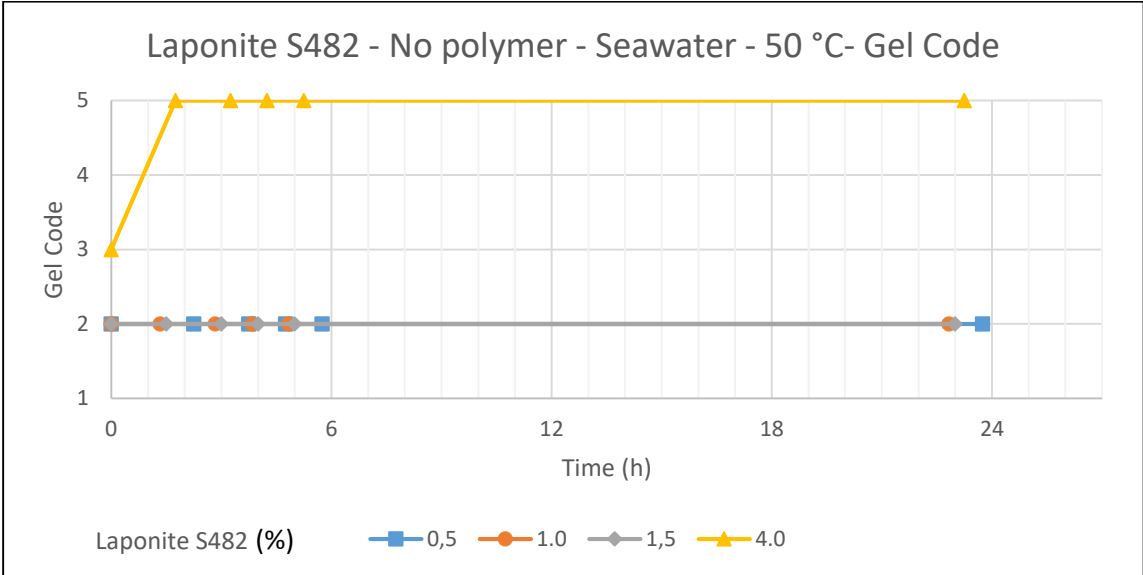


Figure 18. Gel code for gelants with different Laponite S482 concentration in seawater at 50 °C. Note that 0.5, 1.0 and 1.5 % Laponite S482 lines are overlapping.

For the samples containing HPAM, it was not possible to measure a gel code due to the occurrence of flocculation, as shown in Figure 19. As previously discussed in the literature review, the high concentration of ions in seawater can have an adverse impact, leading to flocculation resulting from the aggregation of the structures formed by the surface-edge interactions of the Laponite particles.

This outcome diminishes the feasibility of implementing Laponite S482 with HPAM and seawater. Fluids exhibiting flocculation are unsuitable for injection into reservoirs due to the risk of pipeline plugging. Moreover, there is a high likelihood of ineffective fracture plugging, further reducing the prospectivity of this combination.

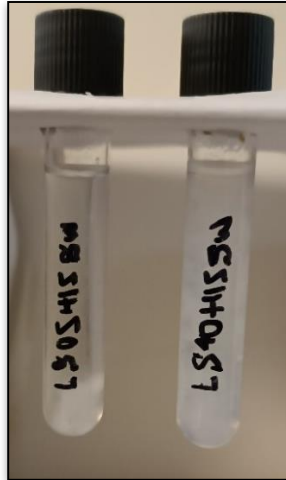


Figure 19. Samples with flocculation. The left tube has 0.5 % Laponite S482 and 0.1 % HPAM in seawater. Right one has 4.0 % Laponite S482 and 0.1 % HPAM in seawater.

4.1.6. Sensitivity to Cations of Laponite S482 Gels

To examine the sensitivity to cations, two specific cations, potassium (K^+) and calcium (Ca^{2+}), were chosen for the study. Brines of potassium chloride (KCl) and calcium chloride ($CaCl_2$) were used to prepare the samples, with a salt concentration of 0.1% w/w, equivalent to typical production brines.

Figure 20 displays the results for samples without polymer, containing only Laponite S482, at both room temperature and 70 °C. It is observed that there is no variation in viscosity for these samples, indicating that neither the presence of potassium cations nor the temperature has a noticeable effect on the behavior of the gels derived from Laponite S482.

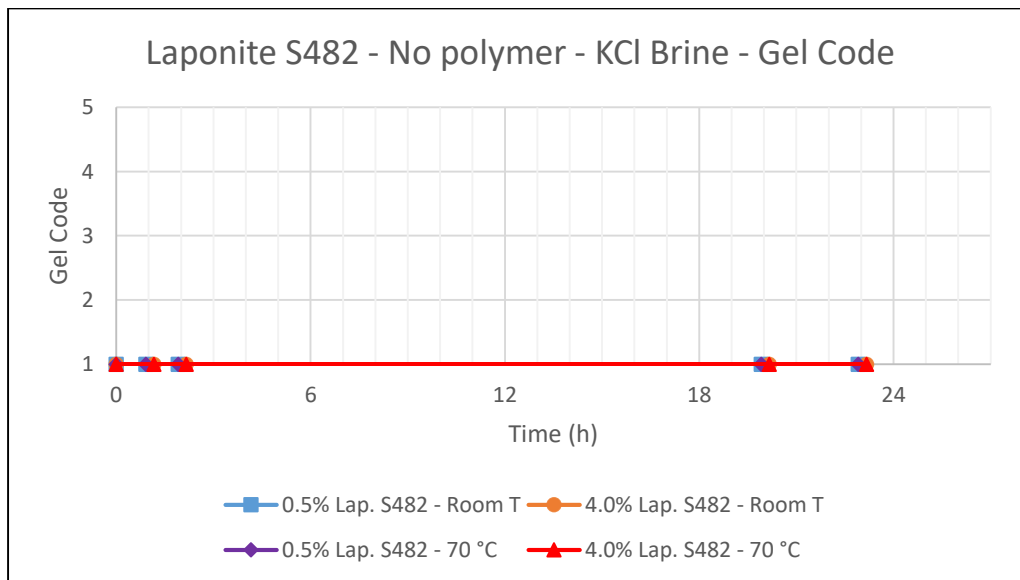


Figure 20. Gel code for gels with different Laponite S482 concentration, using a 0.1 % w/w KCl brine at different temperatures. Note that all the lines are overlapping.

Moving on to samples containing 0.15% HPAM, a slight difference can be observed, as depicted in Figure 21. The addition of the polymer slightly increases the viscosity of the samples. Although the variation is small, it can be attributed to the combined effect of the polymer and the cations. However, once again, temperature does not exhibit a significant impact.

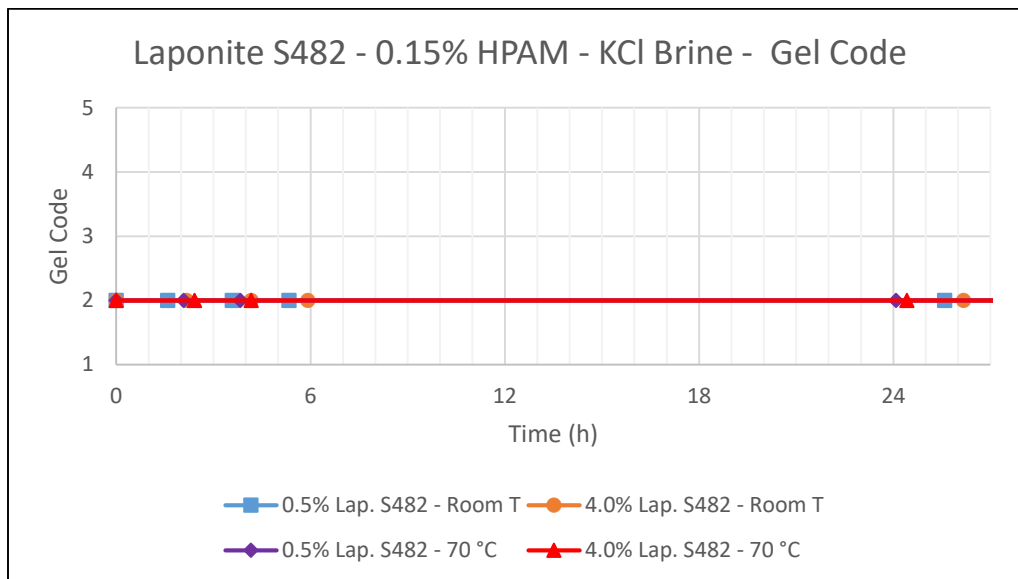


Figure 21. Gel code for gelants with 0.15 % HPAM, varying Laponite S482 concentration, using a 0.1 % w/w KCl brine at different temperatures. Note that all the lines are overlapping.

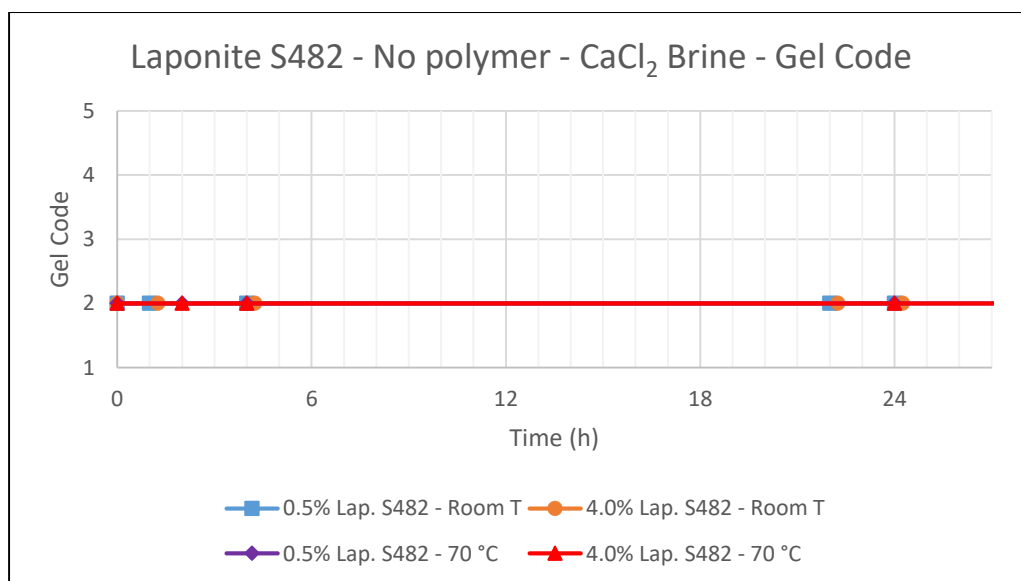


Figure 22. Gel code for gelants with different Laponite S482 concentration, using a 0.1 % w/w CaCl₂ brine at different temperatures. Note that all the lines are overlapping.

Figure 22 presents the results for gelants containing only Laponite S482 with the addition of calcium chloride (CaCl₂). It is evident that calcium, being a divalent cation, has a more pronounced effect to promote gel formation in Laponite S482 systems compared to

potassium. Although the change is minimal, it does affect the viscosity of the samples. However, similar to the potassium samples, temperature does not influence the results.

With the addition of polymer, the changes in viscosity were not significant, resulting in only a minor increase. Figure 23 highlights the similarities between the potassium and calcium samples. However, it is important to note that the gels containing CaCl_2 exhibited slightly higher viscosity compared to the potassium samples, even when the gel code is 2 as well.

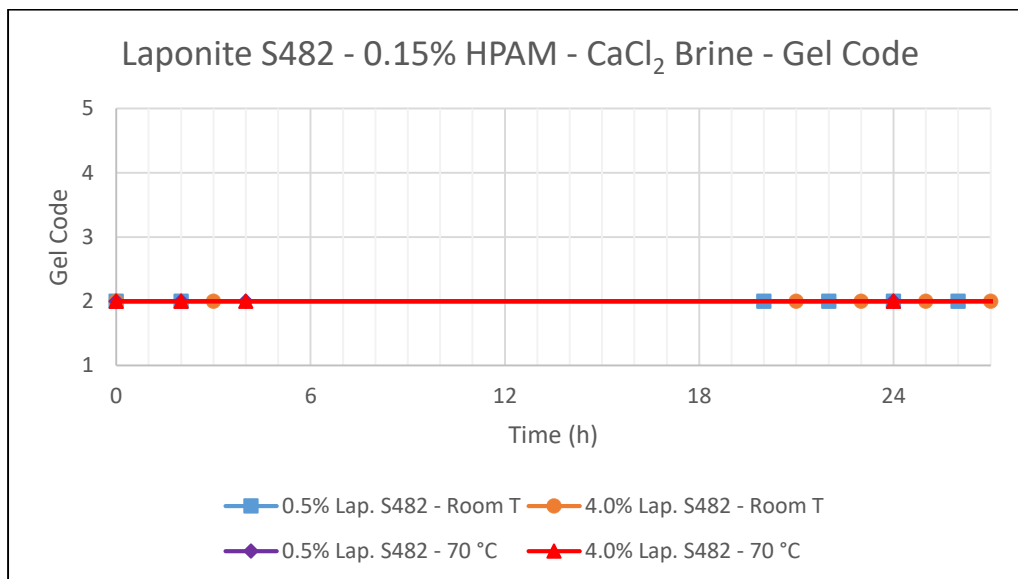


Figure 23. Gel code for gels with 0.15 % HPAM, varying Laponite S482 concentration, using a 0.1 % w/w CaCl_2 brine at different temperatures. Note that all the lines are overlapping.

Overall, these findings suggest that the presence of potassium cations has a minimal impact on the behavior of Laponite S482-based gels, while calcium cations have a slightly more noticeable effect. However, neither the presence of cations nor the temperature variations significantly alter the behavior of the gels when polymer is added.

4.1.7. Chemical Screening of Laponite S482 – CaCl_2 System

The screening of the Laponite - CaCl_2 system was conducted to determine the optimal concentration of calcium chloride (CaCl_2) and Laponite S482 for gel formation. The decision to implement calcium chloride as an additive in the Laponite - CaCl_2 system was based on the understanding that divalent ions, such as calcium ions, tend to yield better results in terms of gel formation. This choice aligns with previous research that has demonstrated the beneficial effects of calcium chloride on the mechanical and thermal properties of Laponite-based products (P. Xu et al., 2020). By incorporating calcium chloride into the system, the objective is to activate the gelling properties of Laponite S482 and enhance its overall performance.

Starting with a fixed concentration of 4.0% Laponite S482, the behavior of the resulting gelant was studied by varying the concentration of CaCl_2 from 0.05% to 0.20% w/w, as shown in Figure 24. At room temperature, no significant change was observed across the

different concentrations of CaCl_2 . All samples remained as stable liquid solutions with slightly higher viscosity compared to water.

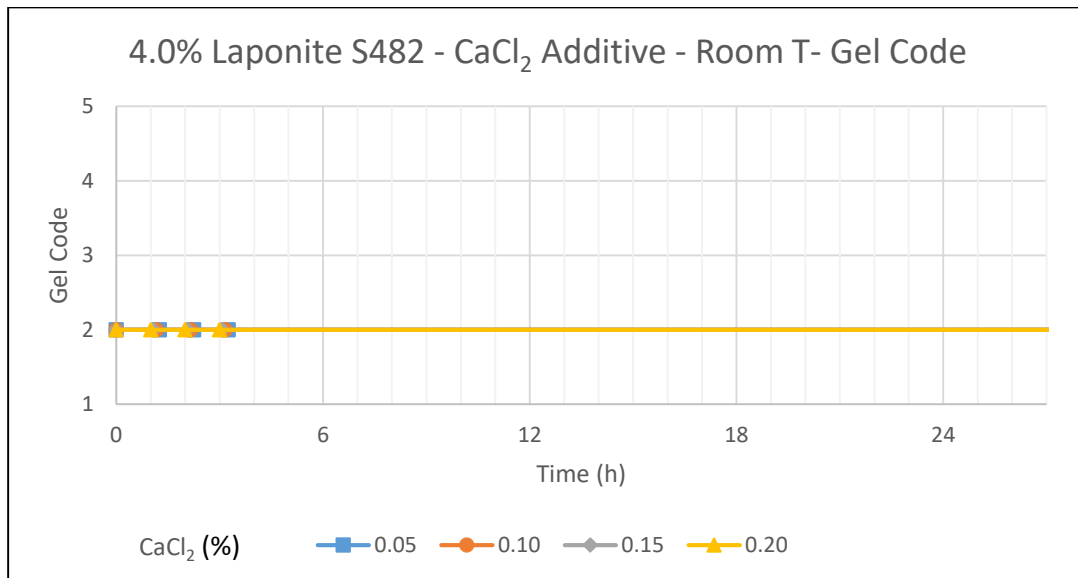


Figure 24. Gel code for gelant systems with 4.0 % of Laponite S482 and varying CaCl_2 concentration at room temperature. Note that all the lines are overlapping.

To evaluate the effect of temperature, the experiments were repeated at 70 °C, as depicted in Figure 25. Only the sample with the highest concentration of CaCl_2 (0.20%) formed a gel after approximately 18 hours, while the rest of the samples remained as stable solutions. Based on these results, the concentration of 0.20% CaCl_2 was selected for further analysis.

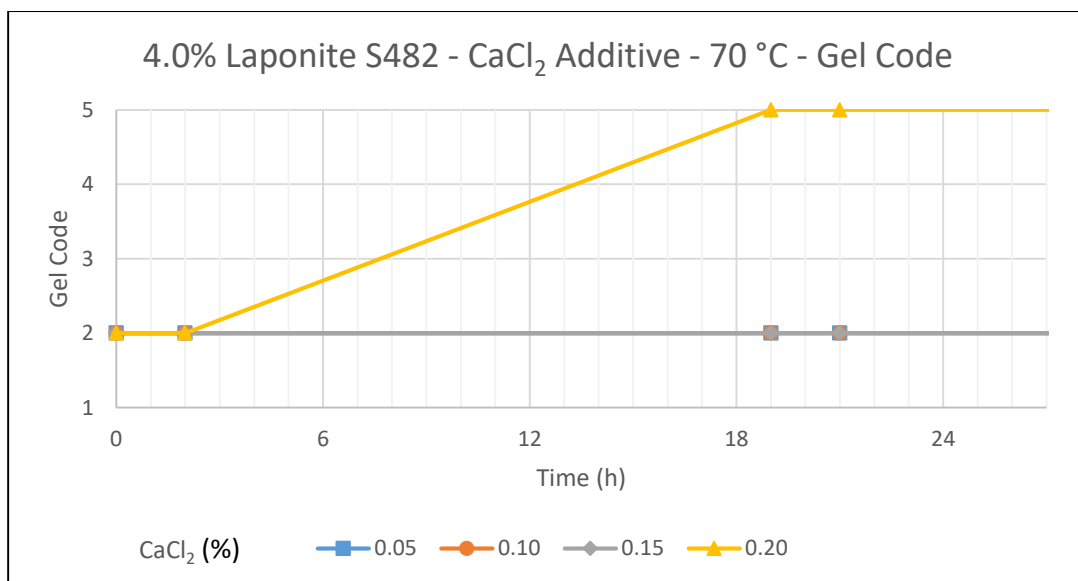


Figure 25. Gel code for gelant systems with 4.0 % of Laponite S482 and varying CaCl_2 concentration at 70 °C. Note that 0.05, 0.10 and 0.15 % CaCl_2 lines are overlapping.

Next, the concentration of Laponite S482 was varied in increments of 1.0% to determine the optimal clay concentration for gel formation. As shown in Figure 26, the sample with 1.0%

Laponite S482 did not form a gel, while the sample with 2.0% achieved the desired outcome and formed a gel at 70 °C after 24 hours.

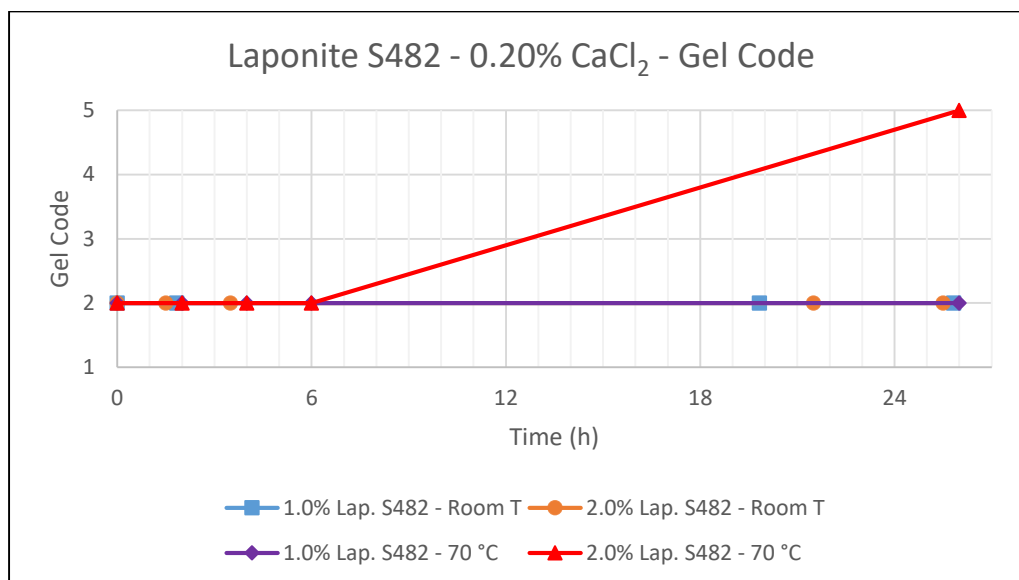


Figure 26. Gel code for gelant systems with 0.20 % CaCl₂ and varying Laponite S482 concentration and temperature. Note that 1.0% Lap. S482 at room temperature and at 70 °C, and 2.0% Lap. S482 at room temperature lines are overlapping.

Based on these findings, the chosen composition for the Laponite - CaCl₂ system is 2.0% Laponite S482 and 0.20% CaCl₂. This gelant remains stable as a liquid solution at room temperature and forms a stable gel at 70 °C after 24 hours.

4.1.8. Chemical Screening of Nanocomposite System

The screening of the nanocomposite consisting of Laponite S482, HPAM, and CaCl₂ involved testing different concentrations of each component to identify the optimal composition for gel formation. It was decided to maintain the concentration of Laponite S482 at 2.0% to facilitate a better comparison of the resulting gels.

During the selection process, some incompatibilities between HPAM and calcium chloride were observed. Low concentrations of CaCl₂ did not lead to gel formation even at elevated temperatures, as shown in figure 27. On the other hand, high concentrations of CaCl₂ resulted in flocculation, which is an undesirable outcome. The presence of high concentrations of HPAM also reduced the acceptable range of CaCl₂ concentration, making it challenging to find an optimal composition.

After careful evaluation, the nanocomposite consisting of 2.0% Laponite S482, 0.15% HPAM, and 0.15% CaCl₂ demonstrated favorable results. This composition exhibited stability as a liquid solution at room temperature and formed a gel at 70 °C after 4 h without the risk of flocculation. This combination of components provides the desired gelling properties for the nanocomposite system.

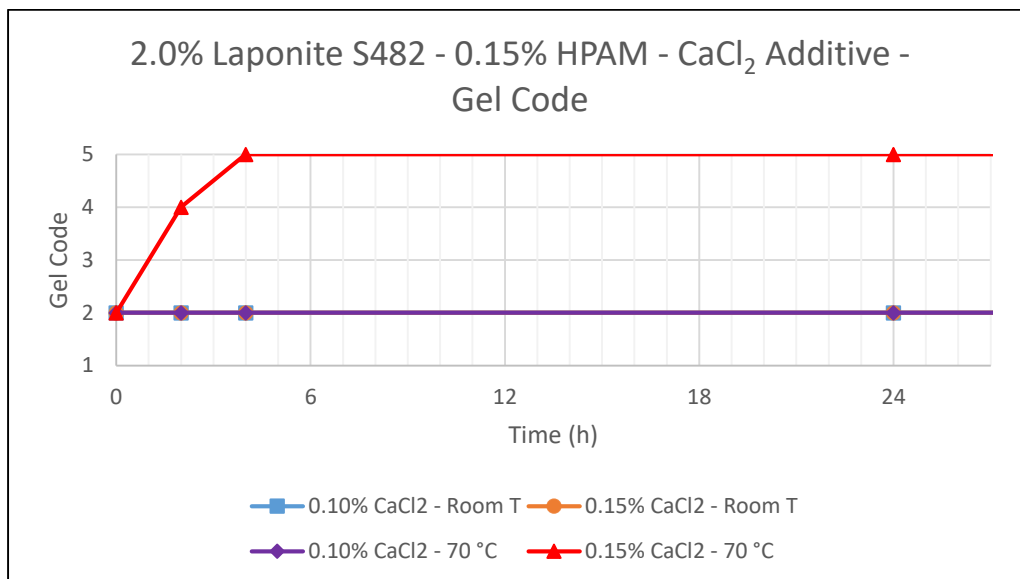


Figure 27. Gel code for nanocomposites with 2.0 % of Laponite S482, 0.15 % of HPAM, varying CaCl₂ concentration and temperature. Note that 0.10% CaCl₂ at room temperature and at 70 °C, and 0.15% CaCl₂ at room temperature lines are overlapping.

4.1.9. Bulk Test Summary and Limitations

Based on the results of the bulk tests, it is evident that gels can be produced from both Laponite RD and Laponite S482. The behavior of the gels with respect to temperature varies slightly depending on the type of Laponite.

For Laponite RD, an increase in temperature accelerates the process of reaching the final state, whether it is a gel formation or simply an increase in viscosity. This indicates that temperature plays a role in the gelation process for Laponite RD.

On the other hand, Laponite S482 generally remains stable as a liquid solution even with increasing temperature. However, it is observed that with a certain concentration of divalent ions, such as calcium chloride (CaCl₂), Laponite S482 can form a gel with increasing temperature. This suggests that divalent ions have a more pronounced effect on the gelation of Laponite S482 compared to monovalent ions.

Indeed, it is important to acknowledge the limitations of the gel code as a measurement of the viscosity or rigidity of gels formed by Laponite. The gel code is a qualitative classification system that provides a rough categorization of the gels based on their visual appearance and behavior. It allows for a quick and convenient assessment of gel formation and stability.

However, for a more comprehensive understanding and accurate characterization of the gels' rheological properties, more advanced techniques such as rheometry should be employed. Rheometry can provide detailed information on the viscosity, elasticity, and other rheological parameters of the gels, allowing for a more precise and quantitative analysis.

By conducting rheological measurements, it would be possible to generate more precise graphs and obtain quantitative data on the behavior of the gels, their flow properties, and their response to different factors such as temperature and additives. This would provide a

deeper understanding of the gelation process and enable more informed decision-making in the design and optimization of gelant systems.

Finally, the following gelant systems were selected after the chemical screening:

- Laponite – CaCl₂ System: 2.0% w/w Laponite S482, 0.20% w/w CaCl₂, de-ionized water
- Nanocomposite System: 2.0% w/w Laponite S482, 0.15% w/w HPAM, 0.15% w/w CaCl₂, de-ionized water

4.2. Filtration Test

The filtration tests were conducted to evaluate the feasibility of injecting the gelants into the fracture model. Three samples were studied, including one sample with only 2.0% Laponite S482 for comparison purposes, as well as two samples containing additional components to enhance their performance as gelling agents. The compositions of the gelants used in the filtration tests were as follows:

- 2.0% Laponite S482
- 2.0% Laponite S482 - 0.20% CaCl₂
- 2.0% Laponite S482 - 0.15% HPAM - 0.15% CaCl₂

The filter with a pore size of 5.0 μm was chosen for the filtration tests based on specific criteria related to the experimental setup and the desired flow behavior of the gelants. The objective was to select gelant candidates that could effectively flow through the fracture model in the core flooding tests and successfully plug the fractures in chalk.

The pore size of the filter was selected to be larger than the average pore size of the chalk (between 0.5 and 5 μm). This ensured that the gelants were not hindered by the small-scale pore structure of the rock matrix. The focus was on evaluating the ability of the gelants to flow through the fracture itself rather than permeating the entire rock formation. The intention was to assess whether the gelants could pass through the fracture and effectively plug it, rather than occupying every single rock pore.

On the other hand, the chosen pore size of the filter was smaller than the size of the glass beads (43 to 52 μm) used to pack the fracture in the core flooding procedure. This ensured that the filter could capture the gelants while allowing the larger-sized glass beads to pass through.

By using the 5.0 μm filter, the filtration tests provided valuable information about the flow characteristics of the gelants through the fracture model. If the gelants were able to flow through the filter without significant obstructions, it indicated their potential to flow through the fracture in the core flooding tests and effectively plug the fractures in the chalk.

In summary, the pore size selection for the filter aimed to strike a balance between allowing the gelants to flow through the fracture model while capturing them for evaluation, without the need to occupy all the rock pores.

4.2.1. Laponite S482 System

As mentioned earlier, this particular sample was conducted to serve as a reference with the two promising gelant samples. The reference does not form a gel at either room temperature

or higher temperatures, as determined from the bulk test results. However, it provides valuable insights for the subsequent tests.

During the filtration test, it was observed that the solution with 2.0% Laponite S482 easily passed through the 5.0 μm filter without any obstructions. However, upon producing a sufficient amount of fluid, approximately 190 g, the filter became clogged, as depicted in Figure 28. This outcome further highlights the behavior of the Laponite S482 system and its interaction with the filter media.

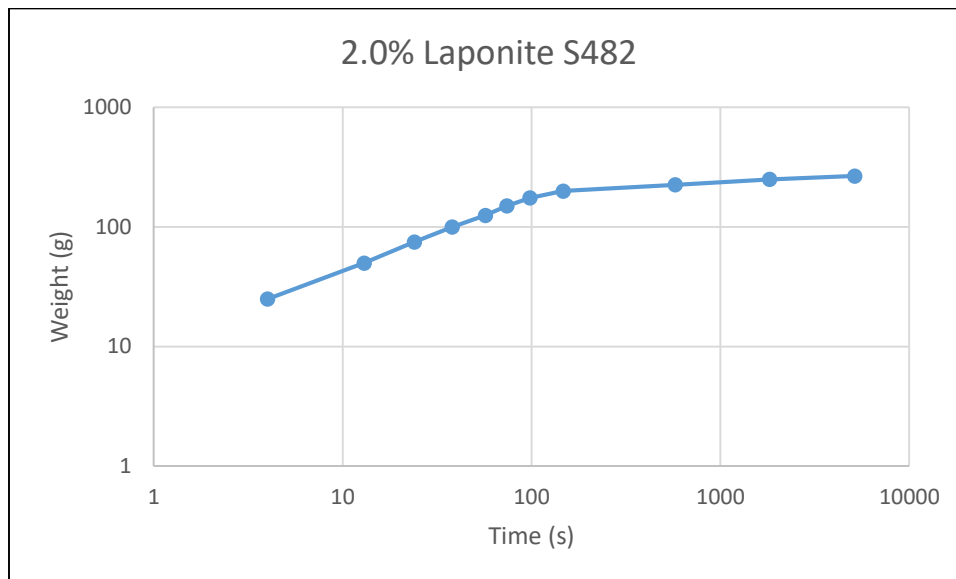


Figure 28. Filtration Test Diagram for Laponite System.

4.2.2. Laponite S482-CaCl₂ System

This section presents the results of the filtration test for the Laponite S482-CaCl₂ system, which was identified as the most promising gelant candidate without polymer. This system consists of Laponite S482 activated with calcium chloride. It showed promise because it remained a stable liquid dispersion at room temperature and formed a gel within the first 24 hours when the temperature was increased to 70 °C.

Figure 29 illustrates the outcome of the filtration test conducted on this sample. It is evident that the filter plugging occurred after approximately 130 grams of fluid were produced, which is a lower volume compared to the previous sample. Furthermore, the production rate was slower compared to the Laponite-only system. This can be attributed to the higher viscosity of the Laponite S482-CaCl₂ system. The addition of cations in the Laponite dispersion enhances the interaction between the clay discs that comprise its structure, resulting in increased viscosity. The higher viscosity, in turn, slows down the flow rate of the sample.

Figure 30 displays the filter cake formed by this sample. The cake exhibited a well-formed structure and measured approx. 2 mm in thickness, which explains the reason for filter plugging. It is important to note that the formation of the filter cake in the filtration test does not necessarily indicate that a similar cake will form during the core flooding tests. The conditions in the core flooding tests are different, and more importantly, the pore size of the

fracture packing is larger, especially considering that the grain size of the glass beads is several times larger than the pore size of the filter used in the filtration test.

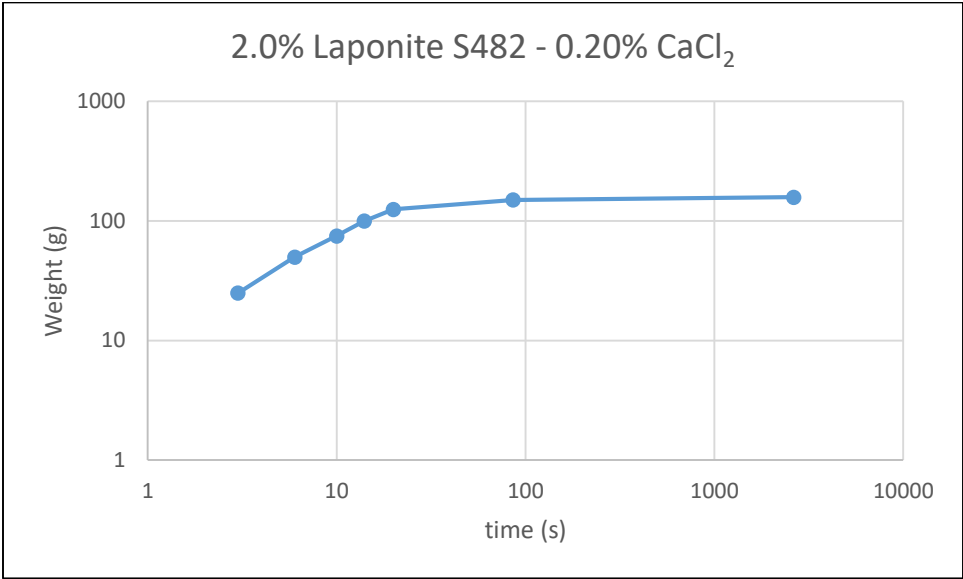


Figure 29. Filtration Test Diagram for Laponite S482-CaCl₂ System.

Therefore, the behavior of the gelant within the fracture model may differ from what is observed in the filtration test. Further evaluation and analysis specifically targeting the core flooding scenario will provide more insights into the performance of the gelant and its ability to plug fractures effectively.

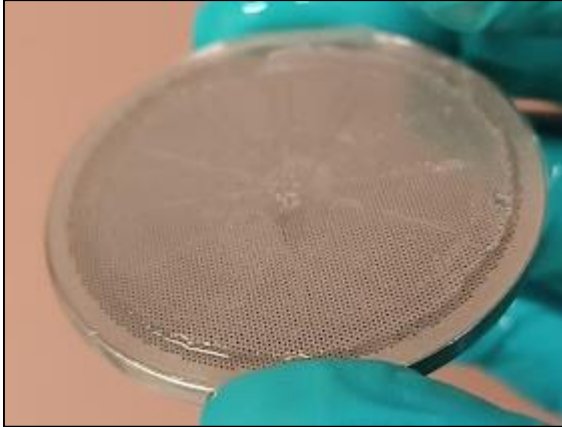


Figure 30. Filtration cake of Laponite S482-CaCl₂ System.

4.2.3. Nanocomposite System

This section presents the results of the seepage test conducted on the most promising nanocomposite system, composed of Laponite, HPAM, and calcium chloride. This particular sample fulfilled the requirements of maintaining a stable liquid dispersion at room temperature and forming a gel at 70°C without any flocculation issues.

Figure 31 illustrates the results of the filtration test for this nanocomposite system. The filter begins to plug at around 80 ml of produced fluid, which is a lower volume compared to the

previous samples (Laponite and Laponite S482-CaCl₂ systems). This outcome was expected due to the visibly higher viscosity of this compound, which also resulted in an increased production time. However, this level of clogging indicates that there would be no significant issues during the injection phase, as the estimated amount of gelant injected during the core flooding test is less than 10 g.

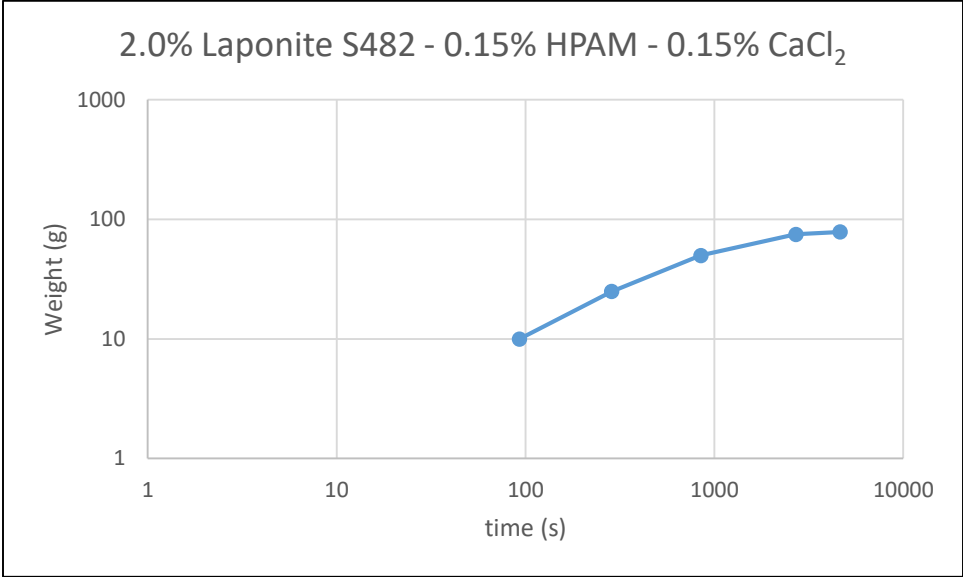


Figure 31. Filtration Test Diagram for Nanocomposite System.

Figure 32 displays the filter cake obtained during this test. The cake appears to be much thinner than the Laponite S482-CaCl₂ sample, measuring less than a millimeter. Additionally, the cake does not exhibit a well-formed structure, as it lacks stiffness and has a somewhat watery texture. It is important to note that no assumptions can be made regarding the core flooding test based solely on these observations, as the conditions and dynamics within the fracture pack may differ significantly. Further analysis specific to the core flooding test will provide a more accurate understanding of the performance and behavior of the nanocomposite gelant system.

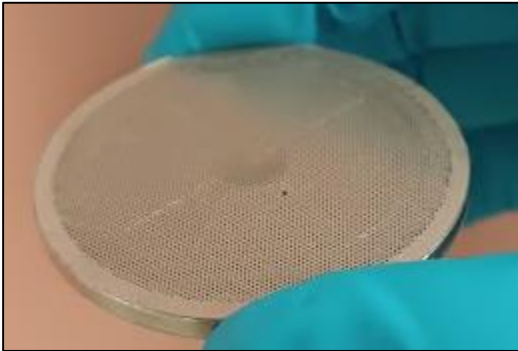


Figure 32. Filtration cake of Nanocomposite System.

4.2.4. Filtration Test Summary and Limitations

Indeed, the filter test has its limitations when it comes to replicating the exact conditions of a fracture pack. The filter used may not perfectly mimic the complex geometry and pore

structure of a real fracture. Additionally, the pore size and permeability of the filter may differ from those of the actual reservoir formation.

Moreover, the injection pressure used in the filter test may not fully represent the higher pumping pressures that would be employed in the subsequent core flooding tests. Higher injection pressures could potentially lead to different fluid behavior and performance in terms of gel formation, flow resistance, and plugging.

However, despite these limitations, the filter test served as a valuable screening tool to identify any potential issues or problems that could arise during the subsequent experiments. It provided an indication of the overall effectiveness and stability of the gelant systems under the given test conditions. The results obtained from the filter test allowed for early identification of any unfavorable parameters or potential problems that could affect the success of the subsequent core flooding tests.

Based on the observations and findings, it was determined that the viscosity of the gelant is a crucial factor in its performance during injection and flood processes. In the case of Laponite solutions, the viscosity is primarily influenced by the structures formed by the surface-edges of the Laponite disks. Higher viscosity indicates larger and more complex structures.

It was observed that samples with lower viscosity had a lower risk of plugging the fracture before gelling. This is because lower viscosity allows for smoother flow and reduces the likelihood of blocking the pores or fractures prematurely. On the other hand, samples with higher viscosity experienced plugging at a lower volume produced during the filtration test. The larger and more complex structures in higher viscosity samples pose a higher risk of blocking the pores before the desired gel formation occurs.

It is important to note that these structures are integral to the gel formation process and are not inherently negative. However, the goal is to ensure gel placement at target.

Both the Laponite and nanocomposite systems can be injected without significant issues. However, the nanocomposite system, due to its higher viscosity, has a relatively higher chance of plugging the fracture before completing the gelant placement. This information highlights the importance of carefully considering the viscosity and gelation properties of the chosen system to optimize the injection and flood processes.

4.3. Core Flooding Tests

The results and analysis of the core flooding tests provide insights into the performance of the gelants in plugging chalk fractures. Two experiments were conducted using different gelants. The first experiment involved a sample containing only Laponite S482 and CaCl_2 , while the second experiment utilized a sample with Laponite S482, CaCl_2 , and HPAM. Both experiments aimed to evaluate and compare the potential of these gelants in plugging fractures in chalk.

The evaluation of gelant effectiveness was based on the Residual Resistance Factor (F_{rr}), which measures the change in water permeability of the core after the creation of the fracture and after the gel treatment. A higher F_{rr} indicates a greater reduction in permeability, indicating a more effective plugging of the fractures by the gelants.

4.3.1. Experiment 1: Laponite S482-CaCl₂ System

This section presents the core flooding tests conducted for the first experiment, focusing on the fracture model and the evaluation of gelant performance.

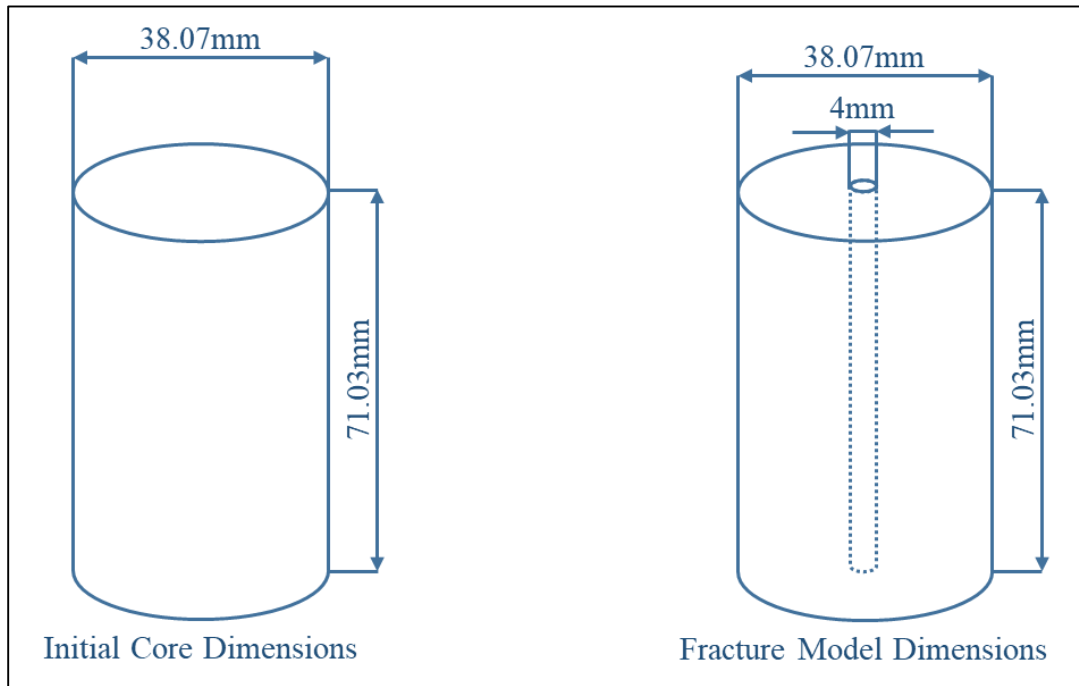


Figure 33. Core Dimensions before and after fracture for core flooding experiment 1.

The fracture model consisted of a chalk core with a diameter of 38.07 mm and a length of 71.03 mm, as depicted in Figure 33. The calculated porosity of the core was determined to be 46.3%. Then, the entire length of the core was fractured with a diameter of 4 mm, and the fracture was subsequently packed with glass beads ranging in size from 43 to 52 μm .

This experiment focused on testing a gelant without the presence of a polymer. The composition of the gelant used in this experiment consisted of 2.0% Laponite S482 and 0.20% CaCl₂ brine prepared in deionized water.

Figure 34 illustrates one side of the chalk core after the fracture was packed just before the gelant injection, as shown in the left photo. The packing material, although different from the chalk, was homogeneously distributed without any empty spaces. This ensured consistent flow of the gelant throughout the fracture, avoiding any variations in its distribution. The right photo displays the same side of the core after the gel treatment. It is apparent that the fracture has been adequately packed with the gelant, indicating successful plugging. The effectiveness of the gelant treatment was further confirmed through permeability measurements.

The results of the permeability tests are presented in Table 4. Prior to fracturing, the permeability of the chalk core was determined by measuring the pressure differentials at distilled water pumping rates of 0.3, 0.2, and 0.1 ml/min. The corresponding pressure differentials (ΔP) were measured as 1650, 1000, and 520 mbar, respectively. These values

exhibited a directly proportional relationship, consistent with Darcy's equation. Using this formula, the permeability value was calculated to be 1.8 md.

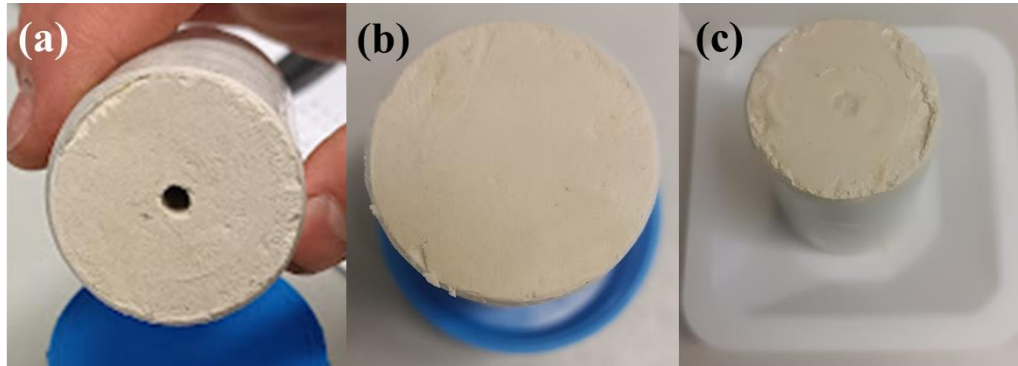


Figure 34. (a) Core Fracture. (b) Fracture Packing. (c) Fracture Plugging by gelant of Experiment 1.

Following the creation of the fracture, the pressure differentials between the core inlet and outlet decreased. Consequently, it was decided to utilize flow rates ten times higher. Once again, a linear relationship was observed between these parameters, resulting in a water permeability value of 43.5 md. Subsequently, the permeability was evaluated after the gel treatment, which led to an increase in the pressure differentials. Therefore, the same flow rates as before the fracture were reinstated. The resulting permeability value was determined to be 3.2 md. The decrease in water permeability indicates the successful plugging of the fracture by the gel. Quantitatively, the residual factor was calculated to be 19.6.

Table 4. Results of Core Flooding experiment 1.

Before Fracture		After Fracture		After gel treatment	
Rate (ml/min)	ΔP (mbar)	Rate (ml/min)	ΔP (mbar)	Rate (ml/min)	ΔP (mbar)
0.3	1650	3	700	0.3	1370
0.2	1000	2	470	0.2	940
0.1	520	1	240	0.1	470
K_w (md) = 1.8		K_w (md) = 43.5		K_w (md) = 2.2	
$F_{rrw} = 19.6$					

4.3.2. Experiment 2: Nanocomposite System

The core flooding test for the second experiment were conducted following a similar process as the first experiment, with the difference being the implementation of a gelant containing a polymer. The composition of the gelant used in this experiment consisted of 2.0% Laponite S482, 0.25% HPAM, and 0.15% $CaCl_2$.

Figure 35 illustrates the dimensions of the chalk core and the fracture model used in this experiment. The core had a diameter of 38.40 mm and a length of 64.34 mm. The calculated

porosity of the core was 47.8%. The fracture, similar to the previous experiment, had a diameter of 4 mm along the entire length of the core and was packed with glass beads ranging in size from 42 to 52 μm .

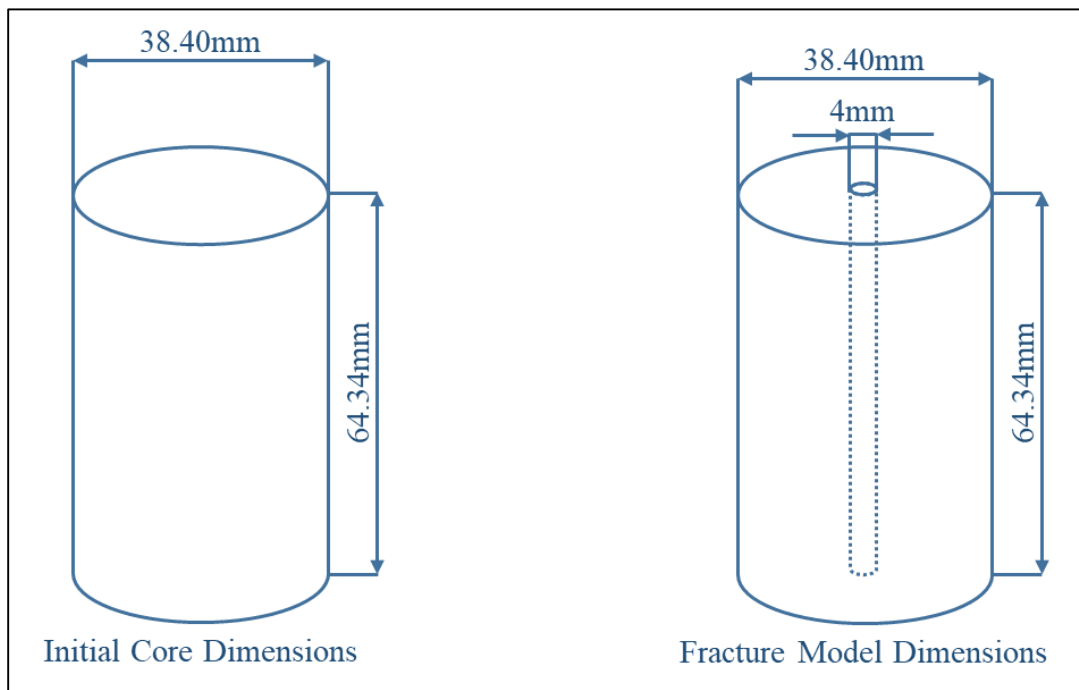


Figure 35. Core Dimensions before and after fracture for core flooding experiment 2.

In Figure 36, the left photo shows the core after fracture without packing, the middle photo shows the core after packing, indicating its homogeneity, and the right photo displays the core after the treatment for fracture plugging with the gelant, demonstrating successful plugging.

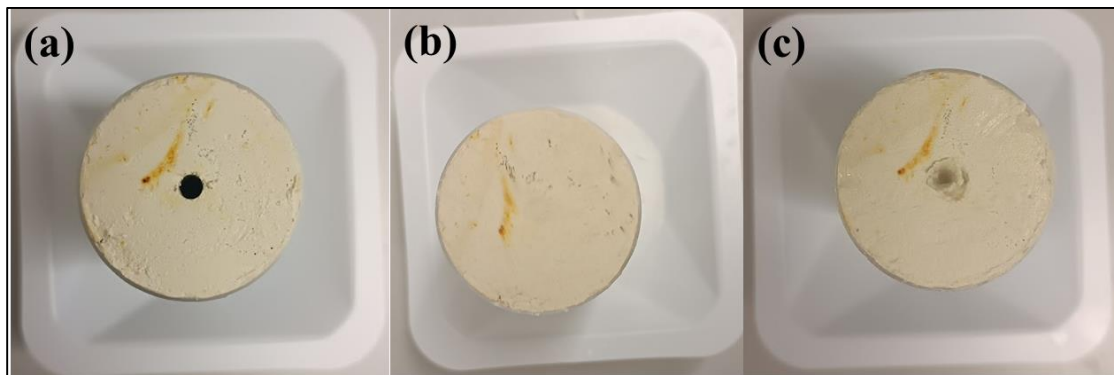


Figure 36. (a) Core Fracture. (b) Fracture Packing. (c) Fracture Plugging by gelant of experiment 2.

To evaluate the efficiency of the plugging, core flooding tests were performed, and the results are presented in Table 5. The calculated permeability to water before fracture was determined to be 1.9 md. After the fracture, due to the presence of glass beads with larger pore sizes, the permeability increased to 24.7 md. Following the treatment with the nanocomposite gelant,

the permeability to water decreased to 4.7 md, indicating effective plugging of the fracture. The calculated F_{rrw} was 5.3.

Table 5. Results of Core Flooding experiment 2.

Before Fracture		After Fracture		After gel treatment	
Rate (ml/min)	ΔP (mbar)	Rate (ml/min)	ΔP (mbar)	Rate (ml/min)	ΔP (mbar)
0.3	1540	3	1230	0.3	640
0.2	1000	2	820	0.2	430
0.1	500	1	420	0.1	210
K_w (md) = 1.9		K_w (md) = 24.7		K_w (md) = 4.7	
$F_{rrw} = 5.3$					

4.3.3. Discussion of Laponite S482-CaCl₂ and Nanocomposite Gel systems

First and foremost, it is important to note that both the Laponite S482-CaCl₂ system and the nanocomposite system exhibit the characteristic of remaining as stable liquid dispersions at room temperature. Throughout the two-week observation period, no visible changes were observed in either system. This is advantageous for their implementation and storage, as there is no risk of premature gel formation prior to injection. Additionally, in cases where immediate use is not required, storage is easier and does not necessitate constant agitation.

The gel formation process differs between the two systems when heated to 70°C. The Laponite S482-CaCl₂ system takes nearly a day to form a gel, whereas the nanocomposite system only requires approximately 4 hours. This implies that the Laponite S482-CaCl₂ system poses less risk during field injection, as slower injection rates can be accommodated if the pumps are not capable of rapid pumping. Conversely, the nanocomposite system may offer advantages in terms of operational time and cost, as it requires less time to transition from injection to resuming operations within the reservoir.

Indeed, adjusting the gel time to achieve an optimal result is an important consideration in gel systems. Different studies, such as the one conducted by (Jia, Yang, et al., 2020), have demonstrated the ability to manipulate the gel time by adjusting the composition, particularly the concentration of initiators or crosslinking agents.

In their case, they used Ammonium persulfate as an initiator and were able to adjust the gel time within a range of 20 to 50 minutes. This flexibility allowed them to tailor the gelation process to specific requirements or operational conditions. In this thesis, exploring the effect of HPAM and CaCl₂ concentrations on gel time could be a valuable avenue of investigation. Since calcium chloride acts as the initiator in this system, it is reasonable to expect that varying its concentration could influence the gelation time. By systematically adjusting the concentrations of HPAM and CaCl₂, it is feasible to achieve gelation within a desired time frame.

It is important to note, however, that while both studies aim to achieve fracture plugging using gel systems, the specific gel formulations and mechanisms involved may differ. (Jia, Yang, et al., 2020) used a different gel system that incorporated a crosslinking agent, specifically N,N'-Methylene diacrylamide (MBA), a toxic substance which is not necessary for Laponite-based gels. Therefore, the parameters and approaches used in their study may not directly translate to your Laponite system.

The viscosity of the gel systems at room temperature is another differentiating factor. The Laponite S482-CaCl₂ system exhibits lower viscosity, making the fluid easier to handle. This was evident during the core flooding tests, particularly during pumping. Higher viscosity typically necessitates higher pumping pressures, as was observed when injecting the nanocomposite system. Eventually, injection was possible until production was achieved, but the pump pressure had increased close to the safety threshold where the pump shuts off. This event did not occur during the first core flooding experiment with the Laponite S482-CaCl₂ system.

Regarding the core flooding tests, both systems successfully reduced water permeability by effectively plugging the chalk fracture. However, the Laponite S482-CaCl₂ system achieved a greater reduction, as indicated by its higher F_{rw} of 19.6, compared to the lower reduction observed with the nanocomposite system, which yielded a resistance factor of 5.3. In this regard, the Laponite S482-CaCl₂ system demonstrates a greater potential for effectively plugging chalk fractures.

There have been other studies that have utilized Laponite for fracture plugging purposes, such as the works conducted by (Bai et al., 2018) and (Tongwa & Bai, 2014). These studies successfully demonstrated the reduction of permeability in flood tests using nanocomposites. However, despite the similarities between these works and the current thesis, there are notable differences in terms of composition. (Bai et al., 2018) used a toxic crosslinker agent mentioned before, MBA, while (Tongwa & Bai, 2014) a less dangerous crosslinker, polyethylene glycol diacrylate, but utilized a hazardous gel accelerator, ammonium persulfate. Additionally, they utilized different types of Laponite, polymers, and additives that are not specifically investigated in this research.

Moreover, another noteworthy study by (Ogienagbon, 2019) employed Laponite RD and HPAM, yielding promising results. However, the main distinction lies in the type of Laponite used and the approach employed during the core flooding tests. In this thesis, the gelant is pumped at room temperature and subsequently heated, while the core itself is already at 70°C during the pumping process, closely resembling the conditions of a real industrial process.

However, it is crucial to acknowledge the presence of certain limitations during the course of these experiments. Firstly, despite my best efforts, it was impossible to guarantee complete packing of the fracture without any areas left unplugged. Additionally, ensuring consistent gelant production posed a challenge, as the gelant appeared colorless and had a viscosity similar to that of water at room temperature.

4.4. Recommendations

The research conducted in this thesis has investigated the application of gel systems, consisting of Laponite and HPAM, as fracture plugging agents in chalk formations, specifically Laponite S482 with CaCl₂ as a promoter gel formation. While the potential of

these gels has been established, there is room for further exploration and in-depth studies to properly characterize and qualify this system. The following recommendations for future work aim to enhance our understanding and effectively assess nanocomposite gels as water conformance agents in fractured chalk formations:

- **Rheology Tests:** Conduct comprehensive rheology tests to measure the viscoelastic properties of the nanocomposite gels. This will provide a better understanding of their mechanical properties, flow behavior, and shear thinning characteristics.
- **Microscopic Analysis:** Perform microscopic analysis to observe the morphology and structure of the nanocomposite gels. This analysis will help verify the presence of Laponite-polymer interactions and provide insights into the gel's internal structure.
- **Characterize Gelation Times:** Carry out more precise and systematic characterization of the gelation times to determine the sol-gel transition phases with greater accuracy. This includes measuring the gelation times in terms of hours and minutes and assessing the rigidity of the gels at different stages.
- **Study Laponite S482-HPAM CaCl₂ Nanocomposite:** Conduct a more detailed investigation of the Laponite S482-HPAM CaCl₂ nanocomposite to develop a gelant that can be tailored to achieve desired gelation times for specific application requirements. Explore the effect of different concentrations and ratios of Laponite, HPAM, and CaCl₂ on the gelation properties.
- **Test with Different Ions:** Explore the effectivity and feasibility of using other additives and ions, especially divalent cations, in the nanocomposite gels. Investigate the influence of different ions on gelation times, gel stability, and mechanical properties to optimize the gel system.
- **Core Flooding Tests in presence of both Water and Oil:** Perform core flooding tests using water and oil to evaluate the behavior and selectivity of the nanocomposite gel systems. Assess their ability to control water flow, improve sweep efficiency, and enhance oil recovery in fractured reservoir conditions.
- **Core Flooding Tests with Different Rock Types:** Conduct core flooding tests using different types of rocks with varying porosity and composition. This will provide a deeper understanding of the sensitivity of the nanocomposite gels to different rock properties and help optimize their performance in diverse reservoir environments.
- **Scaling Up from Lab to Field:** Move from laboratory-scale experiments to field-scale testing to validate the performance and applicability of the nanocomposite gel systems in real fractured reservoirs. Evaluate their effectiveness in controlling water flow and improving reservoir sweep efficiency under field conditions.

Conclusions

This thesis has conducted an evaluation of various gel systems composed of Laponite and HPAM. The assessment encompassed the determination of gel time, interaction with seawater, sensitivity to temperature and cations, injection feasibility, and potential for fracture plugging in chalk. The main conclusions are:

Bulk-experiments:

Bulk tests were performed to evaluate the effect of varying the concentrations of Laponite and HPAM, increasing temperature and adding cations to the gelant systems on their gelation behavior. Through this comprehensive investigation and a chemical screening, two promising systems were identified: one comprising Laponite S482 and CaCl_2 , and the other incorporating HPAM as an additional component to make a nanocomposite. The following conclusion can be drawn from this study

- Laponite RD forms gels easier than Laponite S482
- The increment in temperature makes Laponite RD systems form gel faster
- Laponite RD-HPAM systems increase rigidity with higher concentrations.
- Laponite S482 can form a gel using seawater
- Flocculation is observed when Laponite S482 and HPAM are mixed with seawater
- Divalent cations promote more the gelation in Laponite S482 than monovalent cations
- CaCl_2 can be used as a promotor for gel formation in Laponite S482
- Temperature can be an initiator for gel formation in Laponite S482
- Laponite S482-HPAM nanocomposite are very sensible to divalent cations
- Laponite S482-HPAM nanocomposite are feasible by adjusting HPAM and cations concentration

Filtration Tests:

Promising systems were selected after bulk screening and tested in filtration experiments to secure injectivity of gelant and gel placement in target. Next conclusion can be made from the experiments.

- The viscosity of the gelant system is a crucial factor influencing the potential for fracture plugging. Higher viscosity indicates a greater risk of premature plugging before the gel sets.
- The Laponite S482- CaCl_2 system demonstrated successful pumping during the filtration tests. This indicates its feasibility and potential as a reliable gelant system for fracture plugging.
- Similarly, the Laponite S482-HPAM- CaCl_2 nanocomposite system also exhibited positive results during the filtration tests. This suggests that the gel has the potential to serve as an effective method for plugging fractures, indicating its feasibility.

Core Flooding test:

The potential of two gel systems for fracture plugging in chalk was evaluated and compared with each other. The conclusions of the study are.

- The Laponite S482-CaCl₂ system exhibited a significant decrease in water permeability of fractured model, reducing it from 43.5 to 2.2 md. This reduction corresponds to a high residual resistance factor of 19.6.
- The treatment with the nanocomposite consisting of Laponite, HPAM, and CaCl₂ resulted in a permeability reduction from 24.7 to 4.7 md, corresponding to a F_{rrw} of 5.3.
- The Laponite S482-CaCl₂ system demonstrated better results in terms of reducing permeability and achieving a higher residual resistance factor than the nanocomposite system, indicating its greater potential for effectively plugging chalk fractures.

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