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Lastly, to God almighty, for keeping me alive, hale and hearty to experience all these.

Abstract

Excessive water production is a common challenge the oil industry is faced with. The lifting, treatment and disposal of produced water can cause a financial strain on the profitability of a hydrocarbon asset or even shorten the productive life of the asset. These effects are even more severe in fractured reservoirs as they mature. Among the chemical techniques used for controlling excessive water production, nanocomposite gels (NC) are considered as an effective treatment method. The presence of Nano-clay/polymer network in their structure makes them exhibit stronger fracture plugging potential compared to conventional polymer gel treatments.

 In this contribution, laponite and bentonite NC gels were prepared in deionized and seawater. Their performance was characterised and described. The effect of cations like calcium, and potassium, and also the effect of chalk on laponite dispersions were examined. The performance of various low molecular weight glycols like butyl glycol, butyl diglycol and Polyethylene glycol (PEG) employed as gelation retarders on laponite dispersions were also examined. Finally, core flooding tests were carried out to examine and compare the potential of NC gels as a fracture plugging agent in chalk to laponite gels.

The results showed that laponite generally formed better NC gels than bentonite. Laponite clay also dispersed to form weak to highly viscous NC gels with polymers in deionized water depending on its concentration. The presence of cations helped to screen electro-static repulsion between laponite particles resulting in less aging time and stronger laponite gels. PEG can retard laponite gelation reaction, by adsorbing on the clay surface (steric repulsion) resulting in longer aging time to allow the injection of nanocomposite into target zones before its transformation to a rigid gel. Both NC and laponite gels showed potential for plugging fractures and reducing the permeability of water in chalk. However, NC gels showed higher

resistance residual factor compared to laponite gels. It is proposed that further work should be done to confirm the performance of nanocomposite gels as an effective fracture plugging agent in chalks and also their superiority to laponite gels.

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Nomenclature

OOIP Original oil in place

EOR   Enhanced oil recovery

IFT   Interfacial tension

ET   Total recovery

EMA   Macroscopic sweep

EM   Microscopic sweep

Q   Flow rate

Δp Pressure build/Pressure draw down

WOR   Water/oil ratio

HPAM Hydrolysed polyacrylamide

PEG   poly (ethylene glycol)

NEA   Norwegian environmental agency

WSO   Water shut off

V Crosslink density

M_C Length of chains between crosslinked points

D_{IC} Inter crosslinking distance

M   Mobility ratio

K_{rw} Relative permeability of water

K_{ro} Relative permeability of oil

K_{ab} Absolute permeability

F_r Resistance factor

F_{rrw}	Residual Resistance factor
NC	Nanocomposite gels
NPD	Norwegian petroleum directorate
ϕ	Porosity
K_w	Water permeability
λ	Mobility
F_{rrw}	Residual resistance of water
μ	Viscosity
wt%	Percentage by Weight

Chapter 1 Introduction

1.1 Statement and significance of the problem

Production of excessive water from oil/gas wells is a significant problem facing the petroleum Industry. It is well known that a major by-product related to abundant by-product the production of oil and gas is water (Veil & Clark, 2011); with about three barrels of water being produced for a single barrel of oil (Bailey et al., 2000). Each barrel of produced water amounts to a huge amount of oil left behind in the reservoir; hence, unwanted water production can diminish the profitability of an oil and gas asset. Apart from its negative effect on the profitability on an oil and gas asset, other unwanted water production associated problems such as rapid corrosion of well/surface facilities, fines migration, sand production, scale deposition can shorten the productive lifespan of an oil and gas production asset. Furthermore, poor treatment and disposal of produced water, which usually contains organic and inorganic compounds poses severe environmental risks (El-Karsani et al., 2014).

Water production in oil and gas wells represents an economical, operational, and environmental issue in the petroleum industry, which should be controlled. The presence of naturally induced reservoir heterogeneities in rocks results in the development of high permeability streaks which include fractures, fracture-like features, caves and wormholes which create channels for the flow of unwanted water into the wellbore (Imqam, 2015a). Water may also channel from other sources like leaks in casings/pipes, or due to water coning or a rising oil-water contact. Depending on the origin/type of water production, different techniques are required to tackle them. It is, therefore, vital that the mechanisms of water production be properly understood before an adequate treatment technique can be applied.

Several techniques used to control unwanted water production problems include (Bailey et al., 2000): (1) mechanical solutions involving the use of mechanical packers to seal off troubled zones, (2) completion solutions which require completion operations such as side-tracks or coiled-tubing isolation to isolate troubled zones in more complex cases, (3) Chemical solutions which involves the injection of gels to plug off water production in the troubled zones.

Chemical gel treatments have proven to be a cost-effective and widely used means of controlling water production, especially for in-depth fluid diversions (Imqam, 2015a). Several forms of gel treatments such as micro gels, preformed particle gels, silicate gels,

nanocomposite gels have been presented over the years. The interest in nanocomposite gels for fracture plugging treatments has recently grown due to their ability to withstand harsh reservoir conditions where the use of chemically crosslinked polymers is limited.

1.2   Motivation and Objectives

Excessive water production is a common problem in mature reservoirs (Koohi et al., 2011). As mentioned earlier produced water can diminish the profitability of oil and gas producing assets and in fatal cases lead to early field abandonment. Disposal of this produced water can also pose risks to the environment. Figure 1.1 below shows historical and forecasted produced water and discharged water volumes majority of which are deposited into the sea on the Norwegian continental shelf. Therefore, it is beneficial to reduce the production of water and prevent the use of environmentally hazardous chemicals in the reservoir. In line with the country's policy of reducing the use and disposal of toxic chemicals in the environment, the Norwegian environmental agency provides a list of these chemicals which include lead, Arsenic, Furans, Chromium (NEA, 2014).

The major objective of this thesis is to investigate the feasibility of applying nanocomposite gels based on polymer and clay for fracture plugging in chalk. Although, few studies have been conducted to demonstrate the use of nanocomposite gels for fracture plugging treatments (Bai et al., 2018; Mohammadi et al., 2015), most of these studies involve the use of Chromium (III) acetate or N,N'methylenebisacrylamide as a chemical crosslinker. Some of these studies are also based on the investigation of mechanical and rheological properties of these gels (Aalaie & Marjan, 2012; Haraguchi & Takehisa, 2002a). This thesis will further qualitatively investigate the water plugging capability of Nanocomposite hydrogels in fractured chalk reservoirs. Other secondary objectives of this thesis include:

1. Examine the gel forming potential of different types of clay and polymers
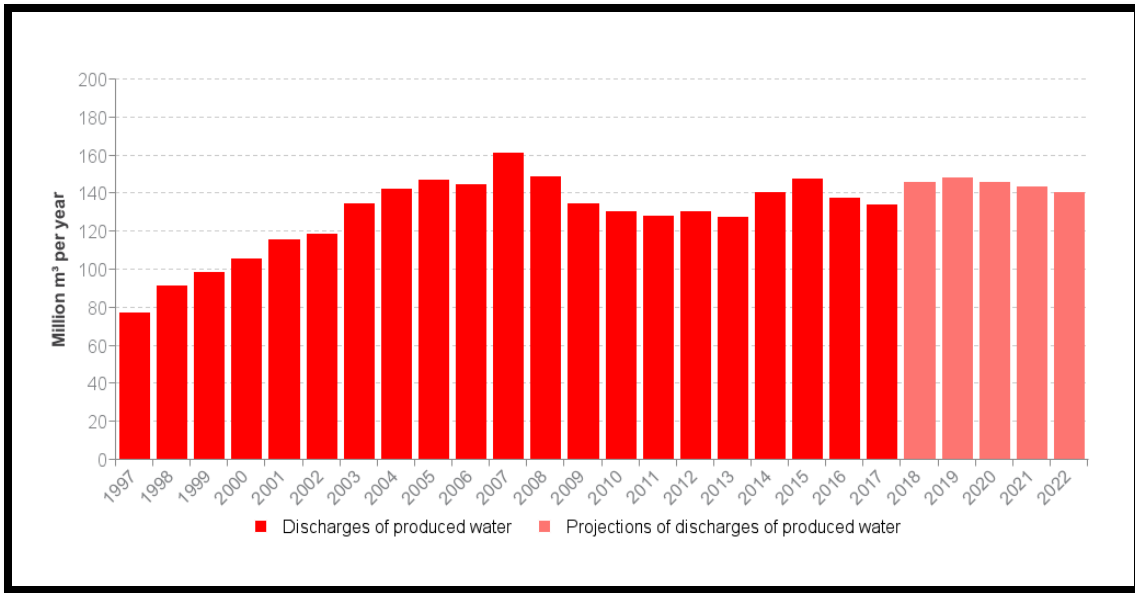
Figure 1.1: Historical and forecast volumes of discharged produced water on the Norwegian continental shelf (NPD, 2018)

2. Investigate the sensitivity of clay to chalk, and cations like calcium and potassium

3. Examine the sensitivity of clay to low molecular weight glycols used as gelation retarders

4. Compare nanocomposite and laponite gels for water plugging treatments

1.3   Scope of work

This thesis is limited to the laboratory bottle tests and laboratory core flood investigation of the potential of nanocomposite gels for water plugging treatments. The work is split into six chapters. Following the introductory chapter 1, Chapter 2 provides a background study into excessive water production. It discusses in detail, the types, mechanisms, diagnosis and several treatment solutions with emphasis placed on chemical solutions. Chapter 3 provides a theoretical review of past researches on nanocomposite gels and factors influencing the success of gel treatments in the reservoir. Chapter 4 presents a description of the experimental procedures and materials that helped achieve the objectives of this thesis. Chapter 5 provides

the results of the experiments, discussions and considerations for further work. Finally, Chapter 6 provides a concise conclusion of the experimental findings.

Chapter 2   Background

2.1   Oil Recovery Mechanisms

Oil recovery mechanisms are broadly classified into primary, secondary and tertiary recovery mechanisms (Green & Willhite, 1998). Primary oil recovery involves the production of the reservoir through its natural pressure depletion. A reservoir's natural pressure may be driven by mechanisms such as water drive, solution and gas cap drive, gravity drainage or a combination of some or all these mechanisms. This recovery mechanism accounts for a recovery in the range of 5-20% of original oil in place (OOIP) (Stalkup, 1983) and it is obviously insufficient for meeting the world's hydrocarbon demands as a lot of oil is still left trapped in the reservoir when the reservoir pressure diminishes.

Secondary recovery processes such as waterflooding and gas injection involve the injection of water or gas to maintain reservoir pressure and displace fluids immiscibly towards the production well (Green & Willhite, 1998). This recovery mechanism accounts for an average oil recovery factor in between 15-50% OOIP (Green & Willhite, 1998). Recovery from reservoirs which have undergone primary and secondary recovery mechanisms typically lie in the range of 35-50% OOIP (Green & Willhite, 1998). Secondary recovery processes like water flooding, however, may fall short of their potential in fractured reservoirs due to the channelling of water through fractures or high permeability streaks. These channels can cause water to bypass oil-rich zones in the reservoir during flooding and trap large amounts of oil behind in the reservoir.

Tertiary recovery mechanisms, also known as Enhanced oil recovery (EOR) methods involve the injection of fluids which are not naturally present in the reservoir to augment the natural drive of the reservoir and ultimately increase oil recovery (Green & Willhite, 1998). This mechanism can be generally classified into thermal, chemical and miscible methods (Van Poollen, 1980). Chemical methods involve the injection of chemicals into the reservoir, e.g. polymers, surfactants, alkaline to increase oil recovery (Green & Willhite, 1998). Miscible methods involve the injection of hydrocarbon gas, inert gas or CO_2 injection under high pressure. Thermal methods on the other hand involve the use of heat to displace oil towards the producing well, this may be through the injection of steam, hot water or the in-situ combustion of oil in the reservoir. The principle of tertiary recovery mechanisms is based on

the interaction of the injected fluids with the reservoir rock and fluids, this may involve the reduction of interfacial tension (IFT), mobility ratio reduction, modification of wettability, reduction of oil viscosity etc. Enhanced oil recovery methods make it possible to increase total oil recovered either by improving microscopic sweep efficiency which involves the mobilization of capillary trapped oil and usually occurs on a pore scale, or by improving macroscopic/ volumetric sweep efficiency.

$$E_T = E_M \times E_{MA} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 2.1$$

Where, E_T - Total oil recovery, E_{MA} - Macroscopic/Volumetric sweep, E_M - Microscopic sweep efficiency. Water channelling problems severely affects an injected fluid's sweep efficiency, which in turn leads to less total oil recovery. Both secondary and EOR methods as promising as they seem are not immune to the negative effects of heterogeneity induced fractures or high permeability zones (Larkin & Creel, 2008). Just as water during waterfloods, injected CO_2 may flow through areas of least resistance through fractures or high permeability zones, bypassing oil in the un-swept zones (Song et al., 2018). This excessive fluid production can lead to low recovery and generally low economics. It is therefore important to find ways to mitigate this problem in order to recover more oil.

2.2   Excessive Water Production

Excessive water production is a major, technical, environmental, and economic challenge in the oil industry (Imqam, 2015a). Globally, about 210 million barrels of water are produced along with every 75 million barrels of oil produced each day (Bailey et al., 2000). From the onset of oil production, water from an underlying aquifer may mix with oil and be produced along with oil. Although the water-oil ratio at this stage may be tolerated if it remains within economic limit and the produced oil is able to cover costs for its disposal. As the reservoir matures water production may become excessive with the production of intolerable amounts of water. "Produced water is a mixture of organic and inorganic components" (Digno, 2019); it requires proper separation and treatment before its disposal. Although the cost of water disposal varies from region to region, it is estimated that more than $40 billion is spent every year for the treatment of unwanted water (Bailey et al., 2000). Aside from the revenue lost in the treatment of this water, other indirect losses may arise due to the loss or delay in production

which is caused by excessive water production related issues e.g. fines migration, mechanical related issues, shut-ins and abandonment, lack of facility capacity (Hill et al., 2012).

2.2.1 Mechanisms of excessive water production

In order to properly tackle excessive water production problems, it is important to identify the source and causes. Produced water may be from natural sources e.g. aquifers or formation water. It may also be from external sources (injected water). Produced water can be classified into three categories: sweep, good and bad water (Bailey et al., 2000). Sweep water is defined as water that helps in the sweeping of oil to the producers, this type of water is beneficial because it aids in the production oil. This includes water from an underlying active aquifer in the reservoir or from water injected which helps to sweep out from the reservoir. Good water is any water that is produced at a rate below the economic limit i.e., oil produced along with this water can pay for the treatment and disposal of this water. Bad water on the other hand is any water whose production is above the economic production limit; this type of water does not aid in the production of oil and oil that is produced with it cannot pay for its treatment and disposal. There are two main problem areas of excessive water production: at the well and in the reservoir (Seright et al., 2001). Each problem area requires a unique type of solution. Therefore, to find optimum solutions to excessive water production problems, the nature and source of the problem must be accurately identified. The scope of this work is only limited to reducing the production of bad water in the reservoir.

2.2.2 Causes of Excessive water production

There are many factors which may lead to excess water production (Imqam, 2015a). Table 2.1 below provides a list of the most common excessive water production causes. While some of these causes may be easily controlled e.g. casing leaks, others require much more complicated approach e.g. fractures.

Table 2. 1 Common causes of water conformance problems, table based on (Imqam, 2015a)

Near-Wellbore Problems	Reservoir-related Problems
Casing leaks	Coning or cresting
Temporary chemical isolation	Fractures, fissures or faults
Lost circulation	Channel from injector
Channeling behind pipe	High permeability streaks
Shut off perforations	Completion near a water zone
Completion into water zone	Watered-out zone

2.2.2.1 Near-wellbore problems

Near wellbore problems can be caused by mechanical problems which may arise as a result of corrosion, holes or cracks around the wellbore or completion problems which may occur as a result of a poor well completion which creating paths for water to channel to the wellbore (Imqam, 2015a). Figure 2.1 shows common near wellbore problems.

Leaks in a casing may provide a conduit for water to migrate from water zones into the wellbore (Jaripatke & Dalrymple, 2010). Water channels behind a casing due to poor wellbore cementing job can also result in water migrating from isolated water-zones into pay zones. Lost circulation may occur during drilling or workover operations when the reservoir fracture pressure is exceeded. Scale or bacteria debris around the wellbore can also serve as flow diverting agents, diverting the flow of water into the wellbore.
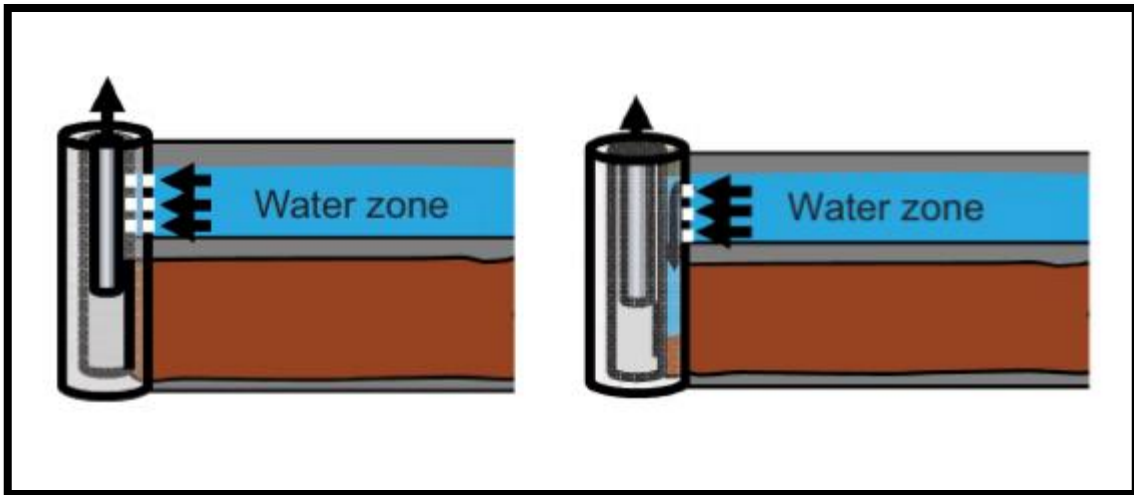
Figure 2.1: Near-wellbore problems- casing tubing or packer leaks (left), water channelling behind a casing (right) (Sydansk, 2011)

2.2.2.2 Reservoir-related problems

Reservoir related problems are more common in mature wells (Imqam, 2015a). Water conning in vertical wells occur in formations with relatively high vertical permeability. Due to pressure depletion around the wellbore, the oil-water contact profile changes into that of a cone which draws water into the well perforations an illustration is shown in figure 2.2. The maximum rate at which oil can be produced through a cone is called the critical conning rate and is usually uneconomical to maintain (Bailey et al., 2000). In horizontal wells this problem is usually referred to as water cresting.

Natural fractures in the reservoir can provide a direct conduit during water floods for water channelling from the injection well to the producer (Jaripatke & Dalrymple, 2010). Extensive fractures may cause injected fluid to by-pass hydrocarbons in the reservoir causing rapid water breakthrough as shown in figure 2.3.
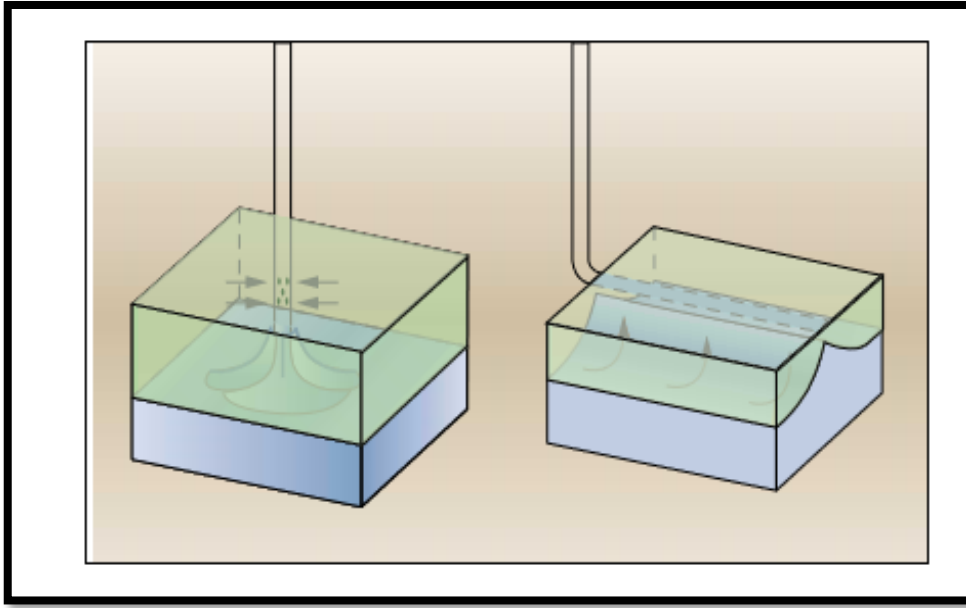
Figure 2.2: Reservoir related problems- water coning in a vertical well (left), water cresting in a horizontal well (right) (Bailey et al., 2000)

The poor design of an hydraulic fracture may cause the hydraulic fracture to intersect a deeper water zone causing an increase in water-oil production ratio (Bailey et al., 2000) as shown in figure 2.4. The presence of high permeability streaks in the reservoir can cause water to flow through theses paths of least resistance causing early water breakthrough and leaving large portions of oil in the reservoir behind un-swept in the permeability zones. Other common reservoir-related water production problems include gravity segregation, water crossflow. There are different causes of excessive water production. Therefore, identifying the right source of a water problem is the first important step in solving this problem.
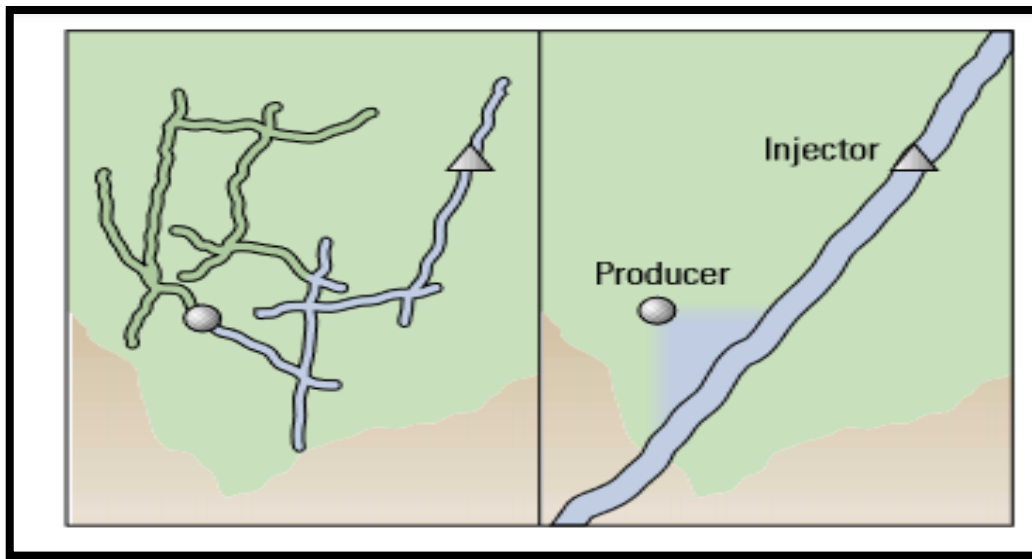
Figure 2.3: Water channelling between injector and producer through fractures (Bailey et al., 2000)
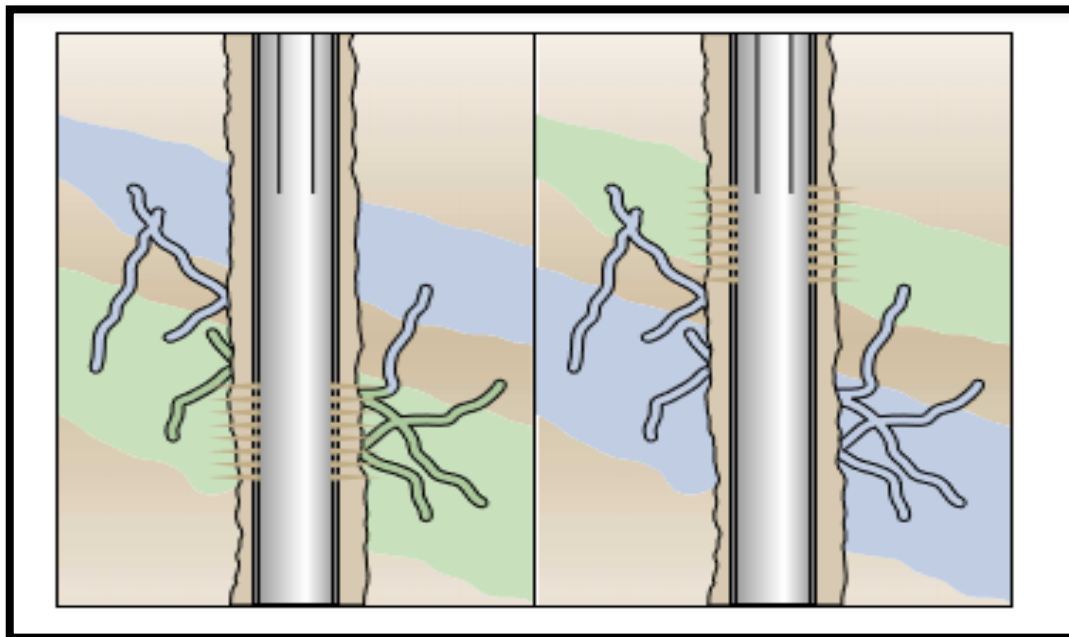
Figure 2.4: Fracturing into a water zone (Bailey et al., 2000)

2.2.3 Diagnostic evaluation of excessive water production

In-order to effectively tackle water problem, adequate diagnosis of the specific source and type of water problem should first be correctly identified (Rabiei et al., 2009). Unfortunately, most producers in the oil and gas industry do not properly diagnose their excessive water production problems which results in the application of inappropriate solutions to water problems and consequently and an overall low success rate of water control problems. The results from well diagnosis can be used to:

- Screening suitable wells for water control
- Identify specific water problem in-order to select appropriate control technique
- Pinpoint the exact water entry location for correct placement of solution.

A study by Seright et al., (2001) has extensively examined different water diagnostic methods. In this study they proposed a straightforward methodology for effective diagnosing excessive water problem. Their study was based on extensive reservoir and completion studies and analysis of many field applications. Before any measure can be taken, the well operator must first determine if there is a water problem. This can be evaluated in three ways: firstly, a sudden increase in water-cut for a certain well or some wells. Secondly, a well or a group of wells may produce at significantly higher water-oil ratios than other wells. Thirdly, a sudden increase in water-oil ratio may be noticed in plots of fluid production vs. time. Another more popular method among large oil and gas operators is the use of reservoir simulation. These numerical systems can help identify possible water problems, evaluate water cut performance, economic-limit rates and even predict maximum water-free rates and breakthrough time. After the operator has confirmed that an excessive water problem does exist, the next steps of action as described by (Seright et al., 2001) is summarized in bullet points below.

- **Leak or flow behind pipe problem?**

If an excessive water problem is confirmed, it should then be considered if the source of this water problem may be from less complicated sources such as casing leaks or channels behind

the casing. Some common methods for diagnosing this problem includes leak tests or casing integrity tests, temperature surveys, noise logs. cement bond logs. Most of the methods previously listed are part of the well routine maintenance tests and as a result, data from this test is readily available. If a leak or flow behind pipe problem is confirmed, the operator can then find suitable treatment solution depending on the exact problem source.

- **Fracture or Fracture-like Feature problem?**

A distinct way of diagnosing fracture problems is to consider whether flow around the wellbore is linear or radial. Flow behind the pipe, fractures and fracture-like features are usually associated with linear flow. While, flow in the reservoir rock matrix is characterized by radial flow. Gel treatments in radial or linear water flow problems differ in placement procedures, volume of gel, and desired properties of the gel. Gel treatments in linear flow problems may be injected without mechanical isolation however, gel treatments must be isolated from hydrocarbon producing zones when dealing with radial flow problems (Seright, 1988). An older method proposed method by Seright et al., (1994) (injectivity/productivity tests) describes the use of Darcy's equation for radial flow Eq. 2.2 for examining flow type. The presence of linear flow is identified by a larger left-hand side of the equation (actual well injectivity/productivity) i.e. about five to six times larger. Consequently, the presence of radial flow is identified when the left-hand side of the equation is lower or equal to the right-hand side of equation. 2.2.

$$q/\Delta p \ = \ \sum kh/[141.2\,\mu \ln(r_e/r_w)] \dots \dots \dots \dots \dots \dots \dots 2.2$$

Where, Q is flow rate, Δp is pressure drawdown and build-up, K is effective rock permeability, h is net pay, μ is viscosity, r_e is the reservoir radius, $r_{w\,is}$ the wellbore radius. Other common methods of distinguishing fractures or fracture like features include core analysis, pulse pressure tests, transient tests, log analysis, inter-well tracer tests, and injectivity/productivity tests.

- **Is there a crossflow compounded matrix problem?**

At the final line of evaluation, once the possibility of other causes of water production problems have been ruled out, the engineer may then deduce the possibility of a radial flow i.e. flow in rock matrix problem. Then the next point of action would be to investigate whether there is a crossflow in reservoir strata. If fluid can crossflow between adjacent strata, then a crossflow can be said to exist. Various methods can be used to investigate the presence of crossflow between layers in the reservoir, common ones include pressure tests between zones, or analysing readily available logs including porosity, permeability, fluid saturation and lithology logs.

2.2.3.1   Water problem plots

Asides, from the method prescribed by Seright et al., (2001), graphical plots are also used in diagnosing various water problems and their sources. These graphical methods provide a quick and visual method of recognizing water problems. Some of these plots are described below (Bailey et al., 2000):

- **Recovery Plot**

A recovery plot is used to detect the presence of water problems, it is presented as a semi-log plot of cumulative oil production versus water-oil ratio (Bailey et al., 2000), see figure 2.5. To determine the presence of water problems the slope of the plot is extrapolated to the economic limit, an extrapolated production value that is equal to the expected recoverable reserves of a reservoir indicates that the well is functioning optimally and there is only production of good water. An extrapolated production value less than the expected recoverable reserve indicates the production of bad water and hence, water control treatments should be considered.
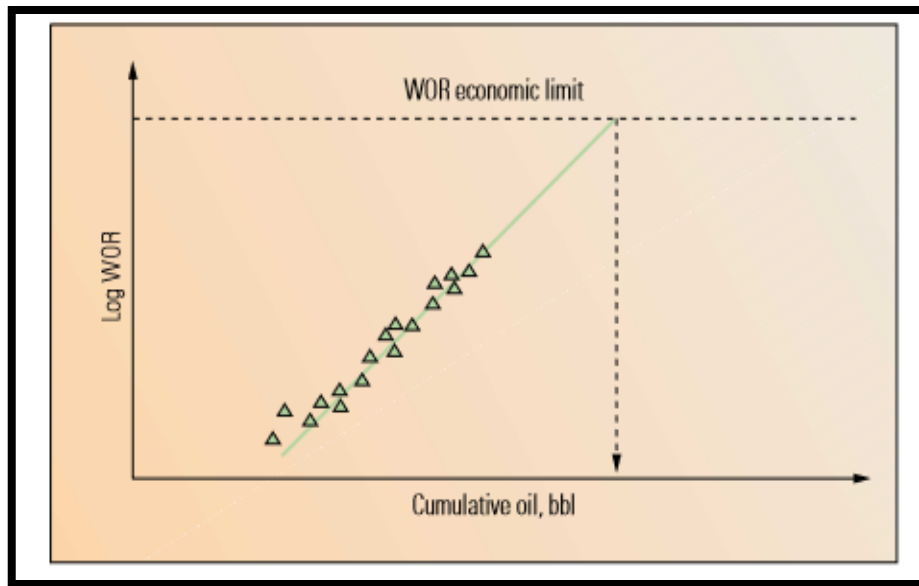
Figure 2.5: A recovery plot showing the logarithm of water-oil ratio versus cumulative oil production (Bailey et al., 2000)

- **Production decline analysis**

Production decline analysis provides a graphical method for analysing declining production rates, and forecasting future reservoir -performance (Agarwal et al., 1998). It also provides a traditional means of detecting water production problems. An increased decline indicates a well problem, which may not necessarily be water e.g. damage build up or severe pressure depletion. Figure 2.6 presents a typical illustration of this plot.

- **Diagnostic plots**

Diagnostic plots are log-log plots of water-oil ratio versus time (Bailey et al., 2000). They provide valuable insight in detecting the presence of a water problem and when combined with other information they can help detect specific source of water problem. Three basic signatures are used to distinguish between the different unwanted water problem mechanisms (Bailey et al., 2000; Chan, 1995). An open flow profile is characterized by a sudden sharp increase in water-oil ratio (WOR), as shown in figure 2.7, this usually indicates that water source is from a fracture, fault or channel behind a casing.
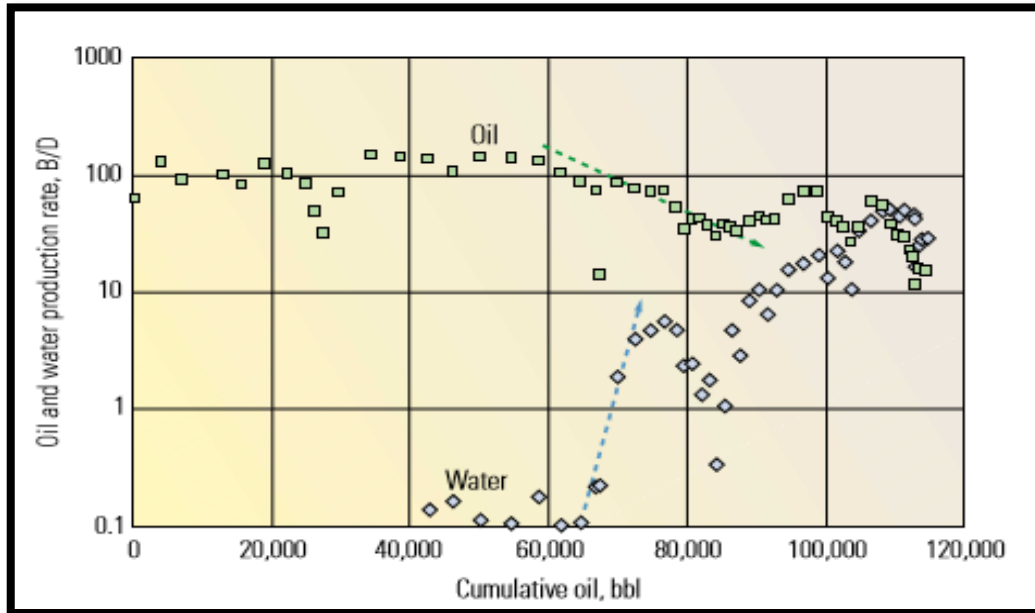
Figure 2.6: A production decline analysis plot (Bailey et al., 2000)

An Edgewater water problem is characterized by a sudden sharp increase in WOR (Bailey et al., 2000); however, this sharp increase is followed by a straight-line curve as, shown in figure 2.8. This curve may have a stair-step profile depending on the permeability contrasts in multilayer reservoirs. Water problems caused by water conning on the other hand, are distinctively characterized by a gentle rise in the WOR curve; this is shown in figure 2.9.
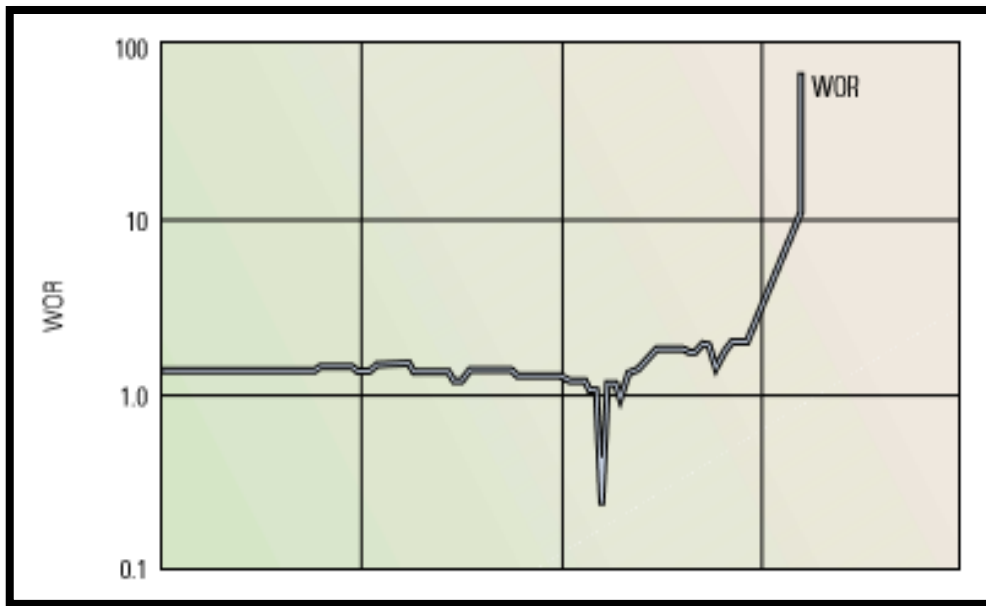
Figure 2.7: A diagnostic plot showing the signature pattern of an open flow water problem (Bailey et al., 2000)
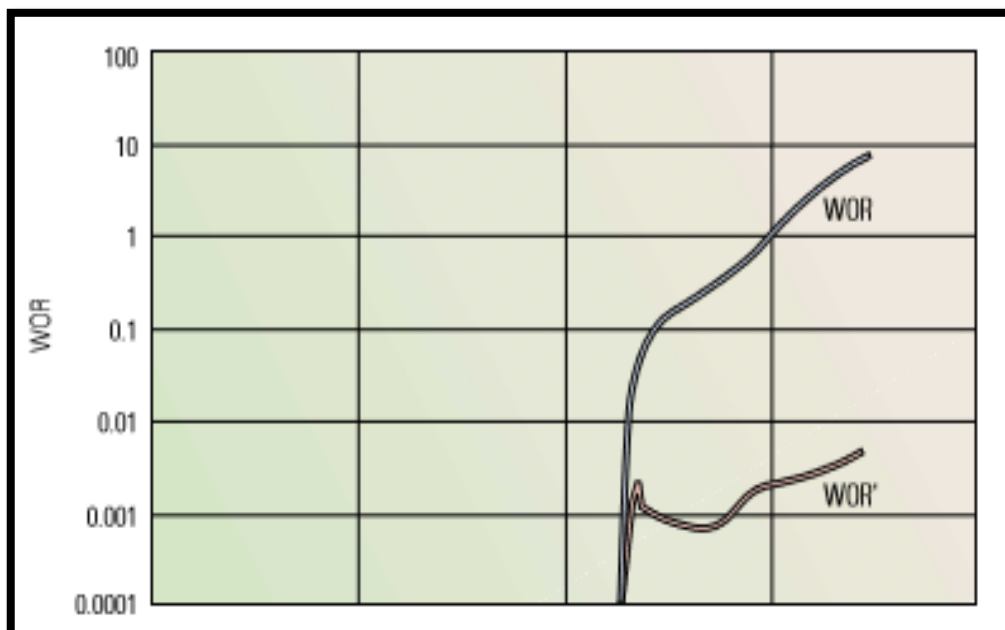
Figure 2.8: A diagnostic plot showing the signature pattern of an edge-water water problem (Bailey et al., 2000)

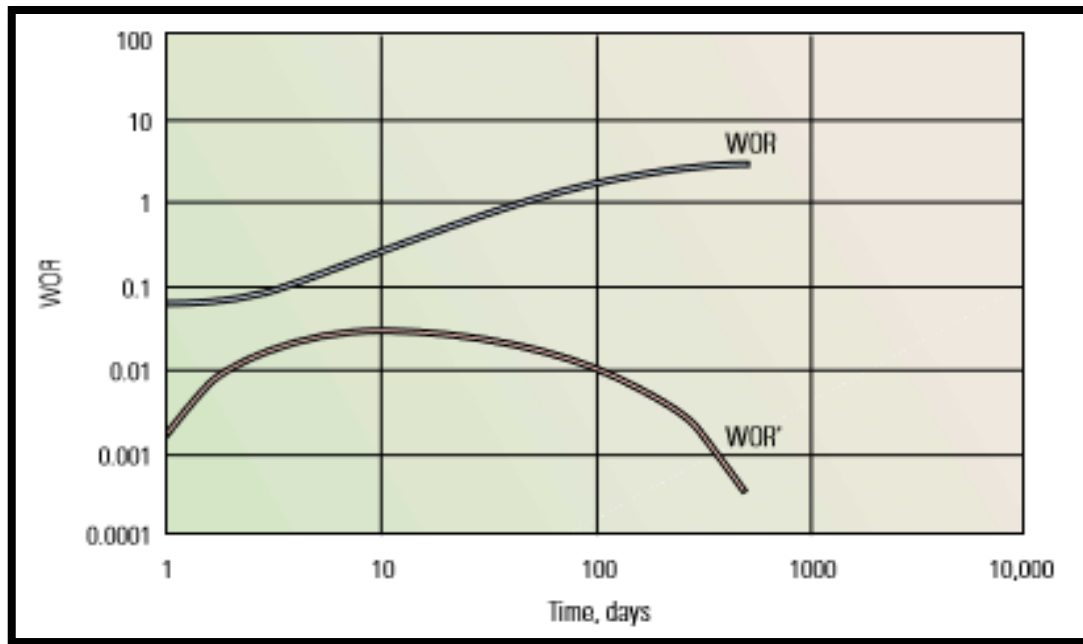*Figure 2.9: A diagnostic plot showing the signature pattern of a water conning problem Bailey et al., 2000)*
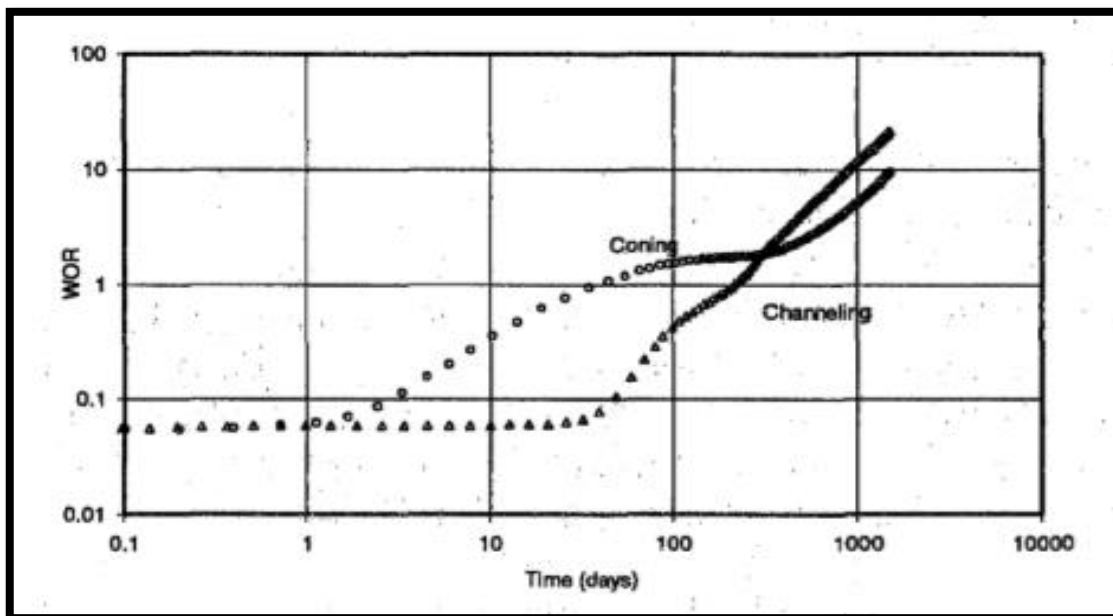

Figure 2.10: A water diagnostic plot showing the descriptive behaviour of water conning and water channelling (Chan, 1995)

It was also proposed that derivatives of the WOR vs time can be used for differentiating whether the excessive water production problem as seen in a well is due to water coning or

multilayer channelling (Chan, 1995). Figure 2.10 provides a plot showing a clear distinction in the shape of two different water problem sources.

2.2.3.2 Well logs

Several well logs can be used to detect water-entry problems (Jaripatke & Dalrymple, 2010). These logs can provide an easy readily available method to detect water problems and their specific type. Common types of these logs include, production logs e.g. Fluid density tool, open hole logs e.g. calliper, casing logs e.g. circumferential scanning tool (CAST), pulse echo tool (PET), cement evaluation logs e.g. ultrasonic-bond log, pulsed neutron logs.

2.2.3.3 Numerical simulators

Well-described reservoir models and reservoir simulation can help provide a means of detecting and even predicting water problems before they happen (Jaripatke & Dalrymple, 2010). They can also help to forecast breakthrough time, water-cut performance and maximum water-free production rates. Recently built advanced reservoir simulators can solve partial differential equations for multidimensional fluid flow to predict the effect of a water-control treatment on reservoir performance and also investigate the efficiency of several placement techniques.

2.2.4 Water treatment placement techniques

When applying water treatment placement adequate care should be taken to select the right treatment placement technique (Jaripatke & Dalrymple, 2010). Placement techniques play a key role in the success of water control treatments, as a result they should be adapted for individual well. Common treatment placement techniques are discussed below.

- **Bull heading:** Bull heading is the most common and economical placement technique (Imqam, 2015a). It does not require the isolation of the targeted zone. However, there is no control over where the treatment fluids might go, this sometimes may result in the sealing

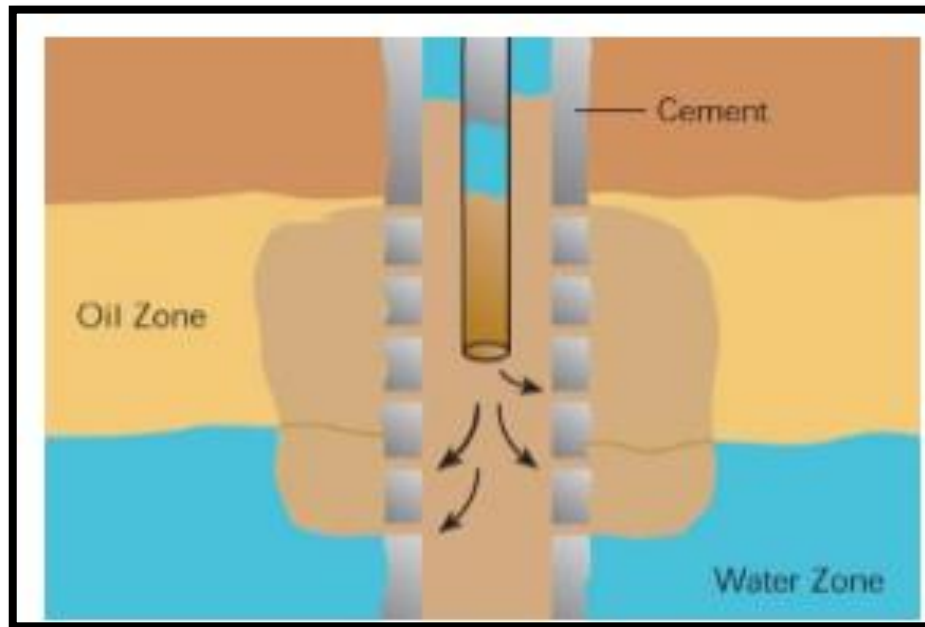of both water and hydrocarbon producing zones. Figure 2.11 shows an illustration of this technique.

Figure 2.11: Bullhead placement technique (Jaripatke & Dalrymple, 2010)

- **Mechanical-packer placement:** To prevent the flow of treatment fluids into oil producing zones, a mechanical packers or bridge plugs can be used to isolate target zones (Imqam, 2015a) (see Figure 2.12).
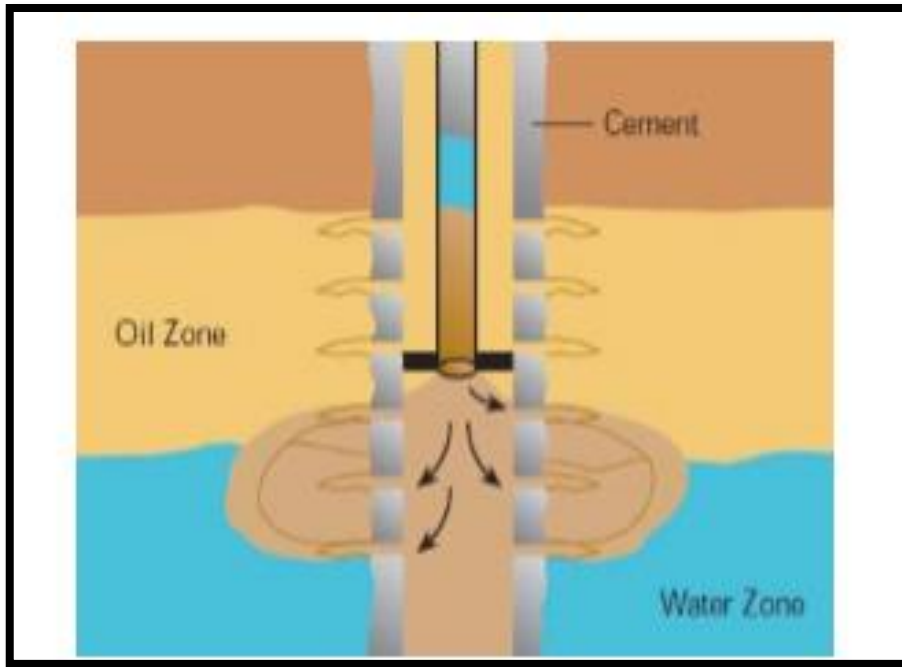
Figure 2.12: Mechanical-packer placement technique (Jaripatke & Dalrymple, 2010)

- **Dual-injection placement:** For a better control of injected treatment fluids, an operator may apply the dual-injection placement technique figure 2.13 below. This technique involves the use of a packer for isolating the zones and the simultaneous injection of the treatment fluid and a compatible fluid down the annulus.

- **Iso-flow injection placement technique:** In Iso-flow injection placement, the treatment fluid is directed into target zones, while, a formation compatible non-sealing fluid which contains a radioactive tag is injected concurrently in the annulus to protect the hydrocarbon-producing zones (Jaripatke & Dalrymple, 2010). An illustration of this set-up is shown in figure 2.14.

- **Transient placement**: Transient placement techniques involves the use of crossflow to help prevent entry into unwanted intervals (Imqam, 2015a), these treatments are injected into the zones that will be sealed

Figure 2.13: Dual-injection placement technique (Jaripatke & Dalrymple, 2010)

Figure 2.14: Iso-flow injection placement technique (Jaripatke & Dalrymple, 2010)

2.3   Water conformance methods

2.3.1   Water conformance mechanism

Water conformance mechanisms refers to all techniques employed in solving excessive water production problems (Sydansk & Romero-Zeron, 2011). It en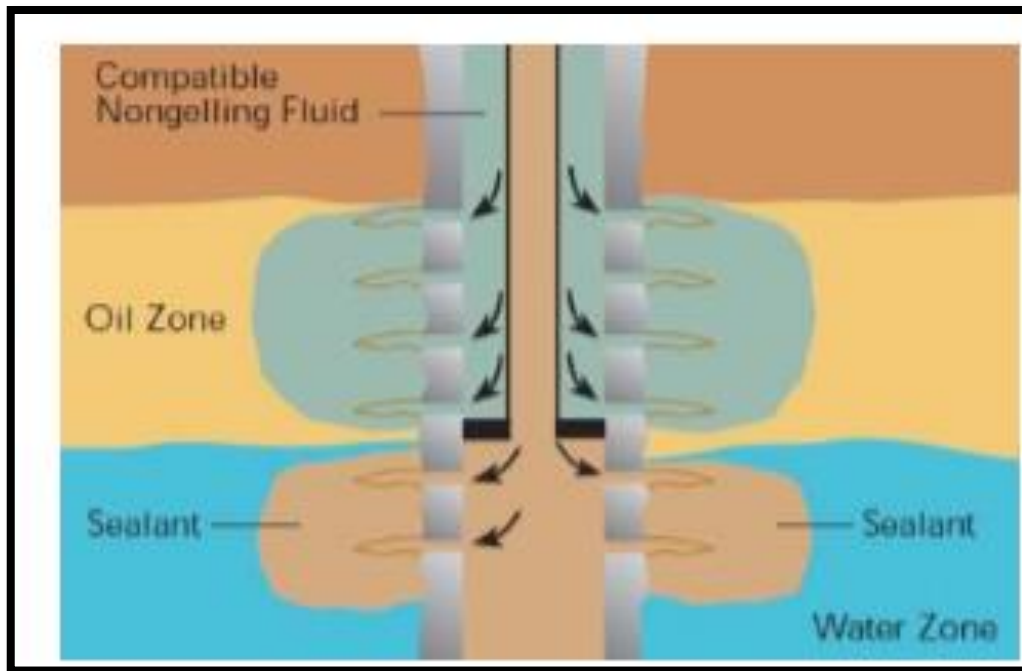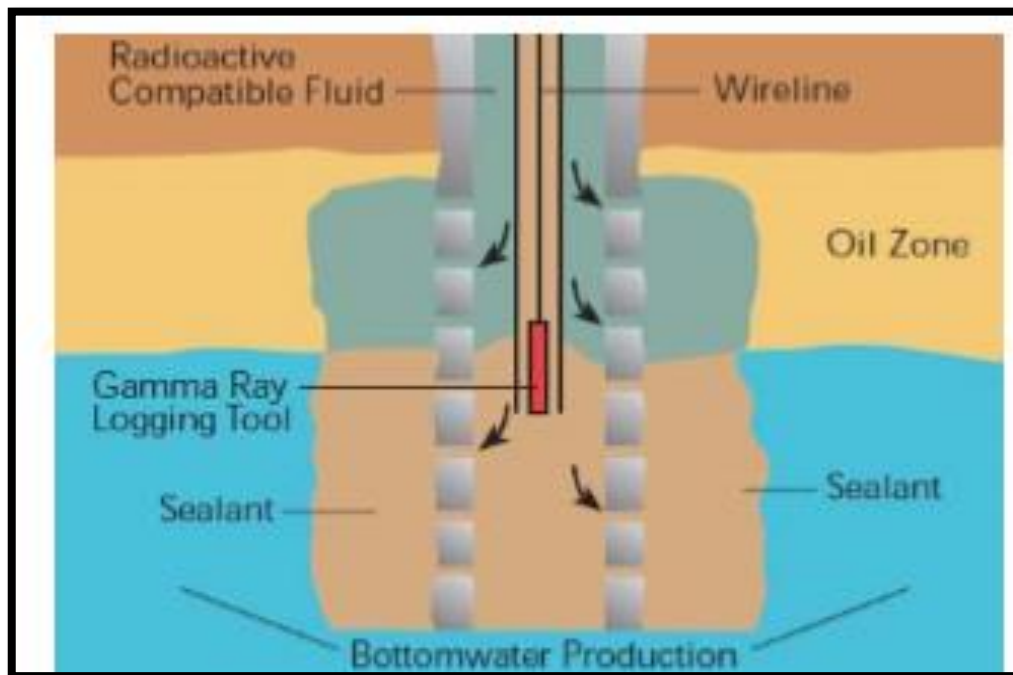compasses all near wellbore and reservoir treatments used in shutting off excessive, deleterious water production. The objectives of water conformance methods include (Imqam, 2015a):

- Reduce unwanted water production
- Improve profitability by reducing water disposal costs
- Improve oil recovery
- Prolong the productive life of an oil and gas assets

Before any water conformance treatment can be applied, it is crucial to accurately determine the type and source of the water problem.

2.3.2   Types of water conformance control treatments

Several types of water conformance control treatments are available. These treatments range from simpler and inexpensive mechanical treatments to other complex re-completion methods. Water conformance control treatments can be classified into three main categories (Bailey et al., 2000): Mechanical solutions, chemical solutions, completion solutions.

- **Mechanical solutions**

Mechanical solutions are preferred treatments for several near wellbore water problems (Imqam, 2015a) such as channels behind casing, rising bottom water and casing leaks. This category of solutions involves the use of a mechanical expandable or non-expandable packer to seal or isolate trouble water zones (Xindi & Baojun, 2017). Expandable packers provide isolation due to their ability to inflate when run into the wellbore and may be retrieved at any point in the life of the well. An example of this is a swell packer shown in figure 2.15 which provides zonal isolation upon contact with some activation fluid in the wellbore e.g. water. Conversely non-expandable packers, although not inflatable and non-retrievable can provide effective sealing of water trouble zones e.g. cement packers.

Figure 2.15: A swell packer (Xindi & Baojun, 2017)
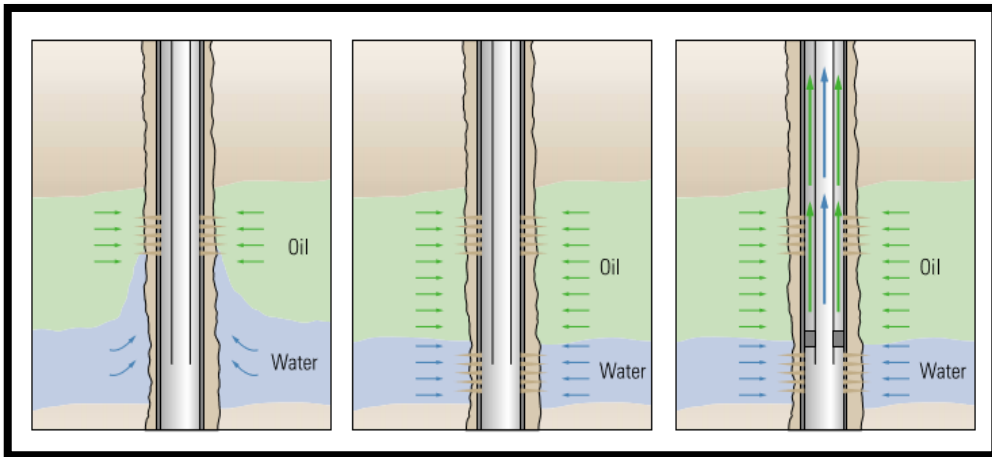
Figure 2.16: Dual completion solutions for solving a water cone problem (Bailey et al., 2000)

- **Completion solutions**

Multilateral well, side-tracks, coiled-tubing isolation, perforation, and dual completion can serves as alternative completion methods to solve more complex water problems such as water coning, incomplete areal sweep, and gravity segregation (Bailey et al., 2000). Figure 2.16 shows a method of using well-recompletion to co-produce water using dual completion tubes.

- **Chemical solutions**

Chemicals solutions can be used to not only treat near-wellbore but also reservoir-related water problems (El-Karsani et al., 2014). There has been a reported large use of chemical water conformance treatments in several fields in China (Bai et al., 2007a). The most common types include, traditional polymer flooding, foam flooding, polymer gels and silicate systems.

2.3.2.1 Traditional polymer floods

Secondary oil recovery processes like water flooding are susceptible to excessive water production problems due to the presence of water channels which causes water to by-pass oil in the reservoir leaving large areas in the reservoir un-swept (Sydansk & Romero-Zeron, 2011). To solve this undesired problem, high molecular-weight polymers can be injected into the reservoir. The addition of polymers into the injected water increases the viscosity of the displacing fluid which creates a favourable mobility ratio between the displacing fluid and the oil and consequently provides better reservoir sweep. Considerable research has been done polymer flooding (Koning et al., 1988; Putz et al., 1995; Wang et al., 2003).

The application of bulk polymer floods should not be confused with the application of polymer gels. There is a clear distinction between conventional polymer floods and polymer gels, while polymer floods require the application of large banks of uncrosslinked polymer solutions, polymer gels in contrast require a much smaller gelant volume and the use of a crosslinking agent (Seright & Liang, 1994). It also should be noted that while the intention for polymer floods is to penetrate deeply into poorly or previously un-swept zones. The intention for polymer gel treatments is to plug the watered-out high permeability zones, while minimizing or totally avoiding penetration in low-permeability zones. Polymer floods suffers limitation on its injectivity. The maximum usable viscosity is typically limited to between three and ten times that of the injection water (Frampton et al., 2004). In addition to this, polymers may also suffer degradation due to shear, salinity, temperature etc. although viscous polymer floods can help reduce water production. It is not a preferred treatment method for most water production problems.

2.3.2.2   Polymer gel systems

Polymer gels are regarded as the most widely used chemical water conformance agents, especially for in-depth fluid diversion. Because of their low cost and ease of injectivity they provide an effective and cost efficient means for water conformance treatment (El-Karsani et al., 2014; Seright & Liang, 1994). The main distinction of gel treatments from traditional polymer floods is the addition of crosslinkers (Abdulbaki et al., 2014). These crosslinking agents promotes the formation of more rigid network between polymer molecules; this enables the formation of a more significant and longer lasting permeability reducing gel. The versatility of their application in both near wellbore and deep-reservoir treatments makes polymer gel treatments a choice for several water conformance problems. Several works have been done on polymer gels systems (Al-Muntasheri, 2008; Al-Muntasheri et al., 2010; El-Karsani et al., 2014; Seright, 1988). Polymer gels may be classified based on their functionality into two categories: sealants and relative permeability modifiers.

Sealants are effective in the complete plugging of water producing zones (Jaripatke & Dalrymple, 2010). This category of gels provides a rigid physical barrier when injected without discriminating between oil and water zones and hence should only be applied in cases where oil and water zones are completely separated. In many mature wells, water producing zones still retain significant volumes of oil and as such it would be beneficial to use relative permeability modifiers. Relative permeability modifiers can reduce the effective permeability of water while maintaining the effective permeability of oil in this trouble zones. This phenomenon is termed Disproportionate permeability reduction (DPR) (Mohammadi et al., 2015). Gels may also be classified based on where gelation takes place (Imqam, 2015a). Based on this, gel treatments may be broadly classified into two categories: (i) In-situ gels, (ii) preformed gels.

2.3.2.3   In-situ gels

In-situ gels are the conventional gel system used for water conformance treatments (Imqam & Bai, 2015b). it involves the injection of a low viscosity gelant into the water troubled zones. This low viscosity gelant is then triggered by some specific mechanism e.g. temperature or pH to form a rigid gel. Developed in the 1970's, the in-situ gelation system provides an efficient means of injecting polymers deep into the reservoir while minimizing the effect of mechanical shear degradation and retention (Borling et al., 1994). Several studies have been carried out on

in-situ gels systems (Avery et al., 1986; Kabir, 2001; Norman et al., 2006; Seright et al., 2001; Seright & Liang, 1994). Gels formed with this process range from weak-rigid gels depending on composition of the gelant and other environmental factors. Recently, newer forms of gel treatments like preformed gels are preferred over in-situ gel treatments, this is because these systems help to overcome several problems associated with in-situ gels treatments like the lack of gelation time control, gelation uncertainty, chromatographic separation of the gelant solution (Chauveteau et al., 2003; Chauveteau et al., 2001; Coste et al., 2000).

2.3.2.4   Preformed gels

Preformed gel systems are formed at surface facilities and injected into the reservoir with no requirement for in-situ gelation (Chauveteau et al., 2003). The new trend of using a preformed gel can help in overcoming several of the drawbacks associated with in-situ gel systems. The common types of preformed gel systems used in the oil industry are listed with their respective developers and field applications in Table 2.2 (Imqam, 2015a). The major differences between all the current commercially preformed gels include particle size, swelling ratio, mechanism of activation and swelling time.

Table 2.2: Common types of preformed gels (Imqam, 2015a)

Name	Developer	Particle Size	Applications
Bright Water®	Chevron, BP and Nalco	Sub-Micro (< 1 μm)	60+ injectors
Microgel	IFP	Micro (1-10 μm)	10+ producers
PPG	PetroChina, MS&T, and Halliburton	Millimeter (10 μm to millimeters)	5,000+ Injectors in China
pH Sensitive polymer	UT	Micro	Not reported

2.3.2.4.1   Preformed particle gels (PPG)

Preformed particle gels (PPG's) are efficient for plugging water problems caused by water channels, fractures or high permeability zones (Abdulbaki et al., 2014). PPG's are highly swelling and super-absorbent polymer gels; they can swell over a hundred-times their original size in liquids. The success of this type of gel treatment depends primarily on its ability to selectively penetrate highly permeable channels or fractures while minimizing penetration into lower permeable previously un-swept zones (Elsharafi & Bai, 2012). Injected PPG gel particles are subjected to high pressure upon injection which causes them to deform and flow through porous media. At distances in the reservoir where pressure is below the threshold pressure, polymer gels swell again creating an effective plug in pore throats increasing residual resistance of high-permeability channels and diverting flow to parts of the reservoir that were previously poorly swept (Abdulbaki et al., 2014). This characteristic of PPG's makes them capable of flowing through porous media even when the particles are larger than pore throats (Bai et al., 2007b) (B. Bai, Liu, Coste, & Li, 2007; Coste et al., 2000; Wu & Bai, 2008). Figure 2.17 shows the swelling characteristics of PPG's with different salinities.

There are some reports that PPG's can only flow through only porous media with high permeability or fractures (Bai et al., 2007b; Liu et al., 2006a). It is also reported that a major disadvantage of PPG is their injectivity; the particle size is relatively large size which prevents them from passing through formations with permeability less that 1D and limits their use to only formations with large permeability contrasts (Abdulbaki et al., 2014). Despite these laboratory scale findings, there have been several successful field applications of PPG's in china. (Liu et al., 2006a) discussed the success of PPG treatment on an injector well in the north Xingshugang region in the Daqing oil field, results showed an economical increase in oil production and a decrease in water production.
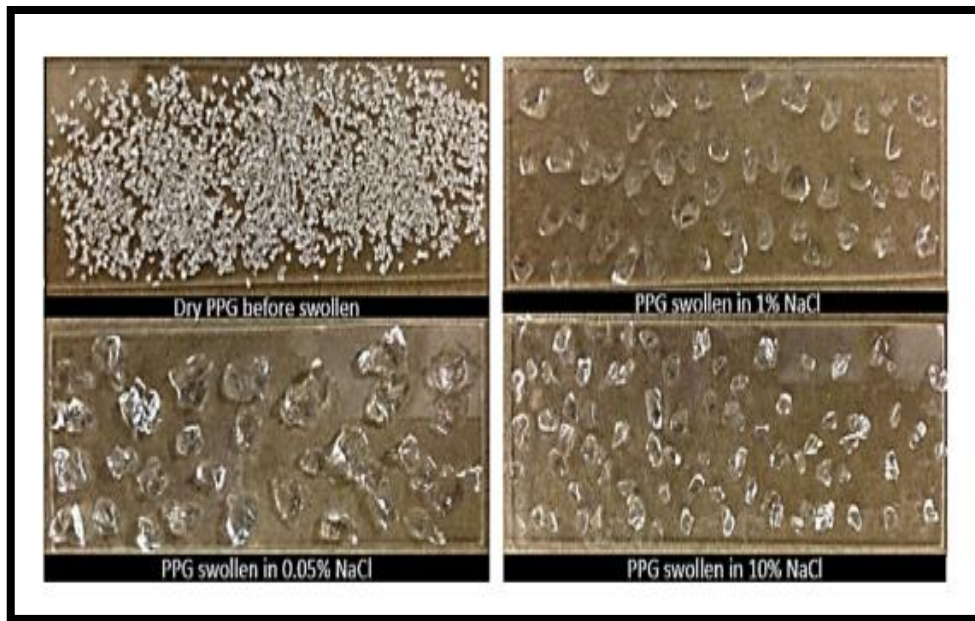
Figure 2.17: Swelling mechanism of preformed particle gel (Imqam & Bai, 2015b)

2.3.2.4.2   pH-Sensitive polymers

pH-Sensitive polymers are one of the most recent polymer gels systems used for water conformance. It was first proposed by (Al-Anazi & Sharma, 2002) when they discovered that gels formed with certain polyelectrolytes, such as polyacrylic acid, are very sensitive to pH conditions. These gels show low viscosity at low pH but can swell up to 1000 their original volume in high pH conditions. The cause of this phenomenon is attributed to the shrinking of polymer chains at low pH, resulting in low viscosity. Contrarily, at the high pH conditions, polymer chains can uncoil due to the repulsive forces between carboxylic groups resulting in an increase in viscosity, an illustration of this process is shown in figure.2.18. Swollen polymer gels serve as effective plugging agent for water conformance control.
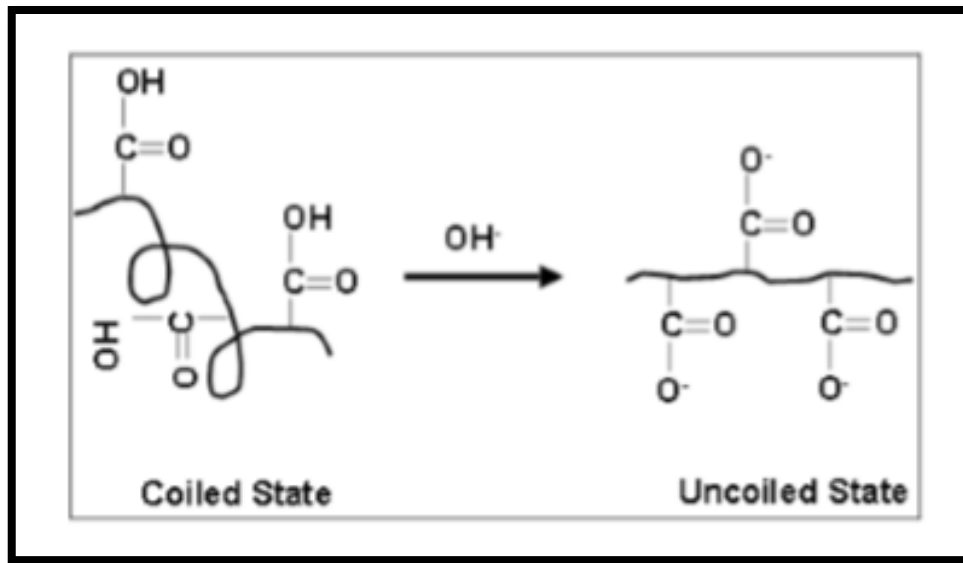
Figure 2.18: Swelling of Polyacrylic acid due to ionization of carboxylic groups (Al-Anazi & Sharma, 2002)

It is observed that the pH-sensitive polymer solutions can propagate further in fractured sandstone reservoirs compared to fractured carbonate reservoirs before gelation (Lalehrokh et al., 2008). This is because pH increases much faster in carbonates due to the presence more acid consuming minerals.

2.3.2.4.3   Bright water

An industry research consortium among BP, Chevron, Texaco and Nalco led to the development of bright water (Abdulbaki et al., 2014). These temperature sensitive gels act as in-depth fluid diverting agents when swelled. Bright water gels are formulated with two crosslinkers: a labile (un-stable crosslinker) and an un-liable crosslinker (Abdulbaki et al., 2014). During application these gel kernels are injected with cold injection water and as the solution propagates through the reservoir, it starts to warm up to the surrounding reservoir temperature until it reaches a pre-determined transition temperature. At this temperature, the liable crosslinker starts to break down, permitting the polymer to absorb water from the surrounding and expand, subsequently plugging trouble zones. This mechanism is frequently compared to the popping of corn kernels into popcorn (Abdulbaki et al., 2014). An attractive feature of bright water is their stability over a vast range of salinities and pH, this is due to its

highly crosslinked nature which makes it less sensitive to harsh reservoir conditions (Abdulbaki et al., 2014; Frampton et al., 2004). Due to the small particle size and low viscosity of the bright water kernels exhibit high injectivity and can propagate long distances deep into the reservoir before they pop (Abdulbaki et al., 2014). There have been several field applications of Brightwater (Frampton et al., 2004; Ohms et al., 2010; Pritchett et al., 2003) results from these projects have shown great potential for the use of Bright water for water conformance. Despite this success, studies have shown that the use of Bright water is not effective in treating fractures.

2.3.2.4.4 Microgel

Microgel as its name depicts refers to the use of size-controlled soft gels with particle size less than 1000nm as relative permeability modifiers and water shutoff treatments (Chauveteau et al., 2003; Chauveteau et al., 2001). Microgels specifically designed for water shutoff (WSO) treatments are non-toxic and fully self-repulsive. They reduce permeability by adsorbing onto rock pore surface and form soft monolayers with a thickness equal to their size. This softness causes high permeability reduction for water without reducing oil permeability. Their size can be exploited during production to cause a permeability reducing effect as desired (Imqam, 2015a). Laboratory tests showed that microgels are mechanically and thermally stable and can be propagated into porous media without any sign of mechanical filtration (Chauveteau et al., 2003). The chemistry of microgels can be chosen to be stable in a wide range of pH and salinity (Chauveteau et al., 2003) which makes the suitable for water shut off and water conformance applications.

2.3.2.4.5 Silicate Gels

Silicate Gels are one of the earliest forms of chemical methods used for excessive water treatments (Stavland et al., 2011). Sodium silicate ($SiO_2:Na_2O$) is an alkaline liquid containing nano-sized particles with pH in the range of 11-13 (Stavland et al., 2011).Upon reaction with an activator, the solution reacts to form a gel which can serve as a plugging/in-depth fluid diverting agent. The gelation kinetics of this system is controlled by the concentration of silicate, concentration of activator as well as temperature and salinity of the makeup water

(Skrettingland et al., 2012). Upon injection and reaction of silicate solution, plugging and permeability reduction ability of this system is established either by formation of an in-situ sol or in-depth filtration of aggregates of size comparable to pore size. Figure 2.19 below shows the sequential steps of polymerization of monomer into large particles.

When pH is reduced, silicate systems can polymerize to form gel (Stavland et al., 2011). On the other hand, at a high pH, the system will remain as solution because the dimer silicate species will dominate the system. There have been several reported laboratory investigations and field application of silicate gel systems in the North Sea (Rolfsvag et al., 1996; Skrettingland et al., 2014) results from these studies have shown great potential for silicate systems as in-depth water treatment agents
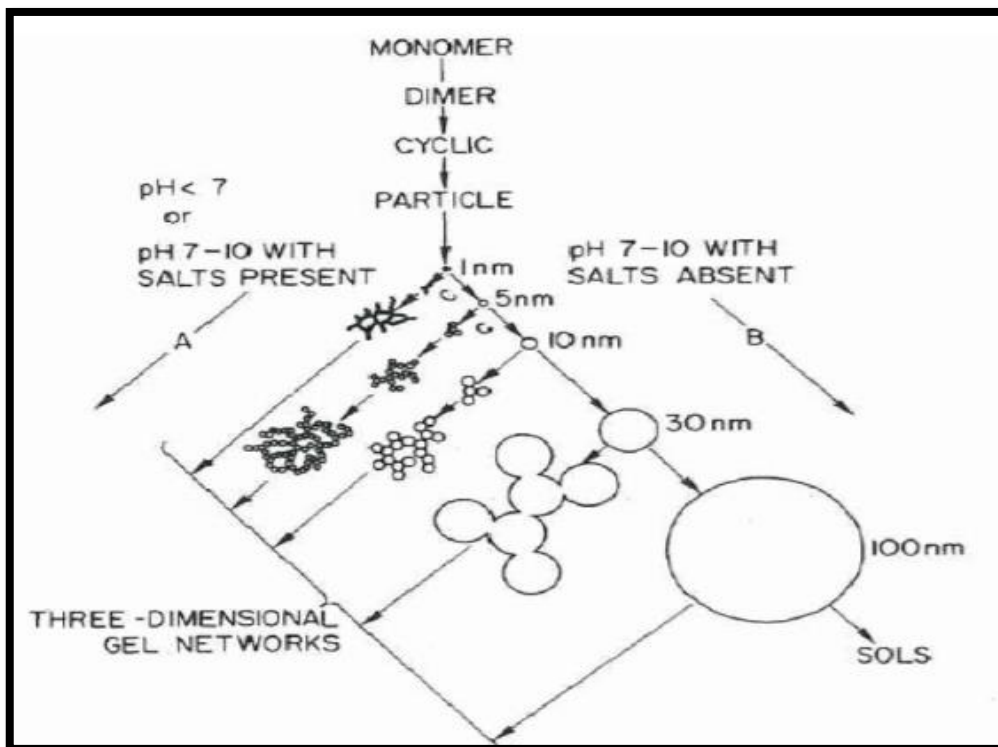
Figure 2.19: Illustration of polymerization of silica (Iler, 1979)

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Chapter 3   Theory

3.1   Chemistry of Nanocomposite Hydrogels

Nanocomposite hydrogels also are polymeric hydrogels capable of eliminating the unattractive characteristics of the conventional polymer gel systems (Haraguchi & Takehisa, 2002a). Nanocomposite hydrogels allow the synergistic combination of favourable properties of both polymer and clay. It involves the use of nano-clays to improve polymer gel properties such as high heat resistance, pressure resistance, deformability (Mohammadi et al., 2015). These nano-clays re-enforce polymer gel network by exfoliating through its matrix and acting as a multifunctional crosslinker which causes the formation of a gel with high resistance against syneresis (Zolfaghari et al., 2006)
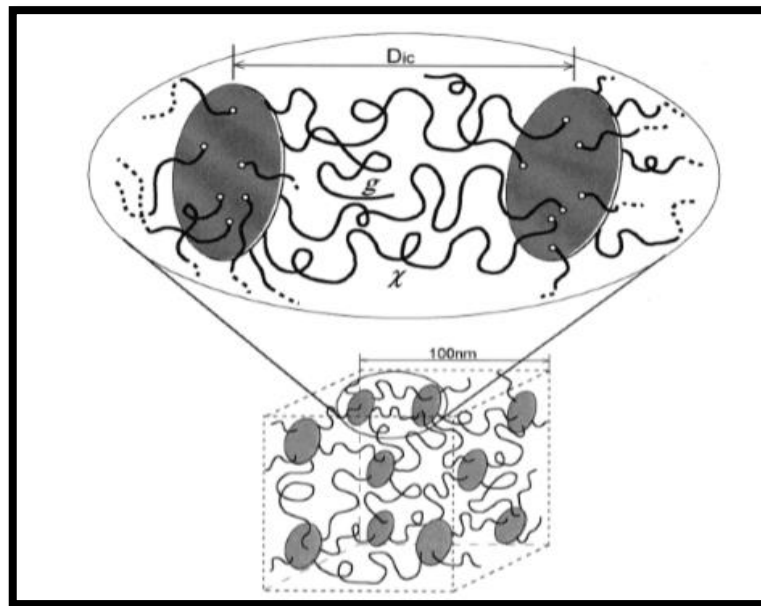
Figure 3.1: Interactions between polymer chains and two neighbouring clay sheets (Haraguchi & Takehisa, 2002a).

Nanocomposite gels are also reported to exhibit higher equilibrium swelling ratio compared to conventional hydrogels without clays (Aalaie & Marjan, 2012). Mohammadi et al., (2015) demonstrated with core flooding experiments that cores treated with nanocomposite gels show

high residual resistance factor and exhibit disproportionate permeability reduction treatments, which makes nanocomposite hydrogels suitable for fracture plugging and water conformance treatments

3.2   Previous researches on nanocomposite-hydrogels

Polymer gels have been used for water conformance treatments for many years (Abdulbaki et al., 2014). Conventional gels used for water conformance are usually polymer and chemical crosslinker based. Polyacrylamide is a well-known water soluble and commercially available polymer used extensively in preparing polymer gels. This is owned mainly due to its chemical robustness, immunity to biological attack, availability and relatively cheaper cost. Crosslinkers used in these gel formulations can be inorganic such as chromium (Cr3+) (Sydansk, 1990), organic such as polyethyleneimine (PEI) (Hardy et al., 1999) or natural such as chitosan (Reddy et al., 2003). Several researchers have examined and discussed these conventional gels in detail (Al-Muntasheri et al., 2009; Chauveteau et al., 1999; Eoff et al., 2007; Seright, 1999; Willhite & Pancake, 2008). Although these gels have enjoyed extensive attention in water conformance treatments through the years. A major challenge has been their susceptibility to degradation when exposed to harsh reservoir conditions. This is owned due to their chemically crosslinked structure which prevents the independent control of the crosslinking density 'v' (number of crosslinked chains per unit volume) and inter-crosslinking molecular weight 'Mc' (the length of chains between crosslinking points).

Haraguchi et al., (2002a) proposed a novel gel formulation called Nanocomposite Hydrogels based on polymer-clay interactions without the presence of any chemical crosslinker. From experimental investigations he found that the use of hydrophilic inorganic nano-clays in polymer gel formulations can help resolve the independent control of the crosslinking density and inter-crosslinking molecular weight; resulting in gels that show high mechanical strength, heat resistance, and ultra-high swelling capabilities compared to the conventional gels. In this novel gel formulation, the inter-crosslinking distance (Dic) is equivalent to the neighbouring clay-clay interparticle distance and can be deduced from the clay concentration as long as the clay is exfoliated, and its sheets are fixed uniformly dispersed locations and when combined with polymer conformations, the inter-crosslinking distance can be converted to the inter-crosslinking molecular weight. Consequently, the polymer and initiator concentrations at a fixed clay content can be used to determine the cross-linking density of the gel. The function

of nano clays used in these gel formulations is to act as a multifunctional crosslinker resulting in polymer gels with high resistance against syneresis (Mohammadi et al., 2015; Zolfaghari et al., 2006).

Haraguchi et al., (2002a) investigated the effect of clay content on the physical and mechanical properties on nanocomposite gels. The performance of different types of clay: hectorite, laponite XLG, Na-montmorillonite on nanocomposite gels was also reported (Zhang et al., 2009). The performance of nanocomposite gels was compared to conventional gels for water conformance was examined (Mohammadi et al., 2015) although, the gelant solution composed of chromium triacetate as a metallic crosslinker, he reported great success with the gel treatment due to their high swelling ratio and low salt sensitivity. The use of low molecular weight non-ionic polymer/nanoparticle dispersed gel for water plugging in fractures was investigated by (Bai et al., 2018). The gelant solution contained a low-molecular weight non-ionic polymer, a chemical crosslinker (N, N'-Methylene bisacrylamide), nano-laponite clay and an accelerating agent. Results from his experimental investigation shows promising potential of the use of this gel in plugging water fractures. Although the previously mentioned gel formulations involve the use of an additional chemical crosslinker, it is strongly argued that the use of chemical crosslinkers in addition to inorganic-clay resulted in gels similar to conventional gels rather than nano-composite hydrogels (Liang et al., 2000).

3.3   Mobility Ratio

Mobility ratio is a dimensionless quantity relating the relationship between the displacing fluid and the displaced fluid during a flooding process. For a water flood operation, in an oil reservoir, it can be written as shown in equation 3.1 (Green & Willhite, 1998)

$$M = \lambda_w / \lambda_o = K_{rw}/\mu_w \times \mu_o / K_{rw} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 3.1$$

Where, M is mobility ratio, λ_w is water mobility, λ_o is oil mobility, K_{rw} is relative water permeability, K_{ro} is relative oil permeability, μ_w is water viscosity, μ_o is Oil viscosity.

Mobility ratio is an important parameter which indicates the condition of a displacement process. It affects both areal and vertical sweep, with sweep decreasing as M increases for a given volume of fluid injected (Green & Willhite, 1998). Mobility ratio is also an indicator of the stability of a displacement process. A mobility ratio greater than 1 (M > 1) can indicate the presence of viscous fingers which can cause water to bypass oil in a water-oil flooding process. Conversely, a mobility ratio less than 1 (M < 1) is considered favourable because it indicates a stable displacement front. The presence of high permeability streaks and lower water viscosity can cause a mobility ratio greater than 1 leading to water channelling and excessive water production (Sydansk & Romero-Zeron, 2011). Water conformance methods aim primarily to shut off water or reduce relative permeability of water in these trouble zone.

3.4   Resistance Factor

Resistance factor is defined as the ratio of mobility of brine to the mobility of polymer/gel in a porous media (Green & Willhite, 1998). The parameter is important in gel/polymer treatment as it helps to characterize gel/polymer behaviour during injection by the magnitude of pressure observed during injection. Resistance Factor can be described in terms of mobility as described below:

$$F_r = \frac{\lambda_w}{\lambda_g} = \frac{k_w}{\mu_w} * \frac{\mu_g}{k_g} \qquad \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \; 3.2$$

It can also be described in terms of pressure as the ratio of pressure drop during gel injection to pressure drop during brine injection

$$F_r = \frac{\Delta p_g}{\Delta p_w} \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots 3.3$$

Where, λ_w is water/brine mobility, λ_g is gel/polymer mobility, k_w is water/brine permeability, K_g is gel/polymer permeability, μ_w is water/brine viscosity, μ_g is polymer/gel viscosity, Δp_g is pressure drop during gel injection, Δp_w is pressure drop during brine injection.

3.5   Residual Resistance Factor

Residual resistance factor is defined as the ratio of water or oil mobility before polymer/gel injection to the ratio of water mobility after polymer/gel injection. This parameter

describes the magnitude of permeability reduction caused by the injected polymer/gel. It can be written as:

$$F_{rr} = \frac{\lambda_w}{\lambda_{wp}} \ \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots.3.4$$

Because water/oil viscosity remains the same, this parameter can be eliminated so equation 2.6 can be simplified as written below in equation 2.7.

$$F_{rr} = \frac{k_w}{k_{wp}} \ \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots.3.5$$

At any given injection rate, F_{rr} can also be expressed as the pressure drop during water/oil injection.

$$F_{rr} = \frac{\Delta p_{wp}}{\Delta p_w} \ \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots.3.6$$

Where, F_{rr} - Residual resistance factor, K_w -water permeability before polymer/gel treatment K_{wp} is water permeability after polymer/gel treatment, Δp_w is pressure drop during water injection before polymer/gel treatment, Δp_{wp} is pressure drop during water injection after polymer/gel treatment. Equations 3.4, 3.5 and 3.6 can be written in terms of oil. A good in-depth relative permeability modifier should have a high-water residual resistance factor, while maintaining a low oil residual resistance factor.

3.6   Gel Strength

The elastic strength of a gel indicates the resistance of the formed gel to physical deformation that the gel will encounter while extruding through a constriction in its flow path (Sydansk, 1990). After the preparation of Nanocomposite hydrogels at the surface, they would be injected into the formation. It is required that formed gel should be able to withstand the high-pressure gradient at the near wellbore vicinity which gets lower at distances further from the wellbore. After the injection of gel, the well will be shut-in for a period time to allow sol-gel transition. A sol is a low viscosity colloidal solution, while a gel is a high viscosity colloidal solution. After this shut-in time, the formed gel should be strong enough to withstand injection pressure of when subsequent flow is resumed.

3.7   Gel Syneresis

Syneresis refers to the process by which polymer gels after some time tend to contract thereby expelling the solvent phase (Zhang et al., 2015). This process has an important impact on the overall effectiveness of a gel treatment. However, in this study we investigate the use of nanocomposite hydrogels which have been reported to have high resistance against syneresis (Zolfaghari et al., 2006) therefore, the effect is not considered in this study.

Chapter 4   Experimental Procedures and Materials

Experimental procedures and materials used in this thesis are presented in this chapter. This chapter is divided into six sections, and an outline of various sections is given below:

1. Firstly, the different types of polymers and clays used in this research are presented

2. Secondly, an experimental procedure for tube testing of the various polymer and clay solution is presented as well as the equipment used

3. Subsequently the most promising candidate selected from previous step is the qualitatively analysed for its chemical interactions with cations and gelation retarders

4. In the next section, a core flooding set-up is presented, type of core, properties, brine and oil properties are presented

5. Core flooding procedure is presented, gelant injection, shut-in time and temperature, and fracture plugging ability is examined

Figure 4.1 presents a schematic diagram of the experimental steps followed in this study
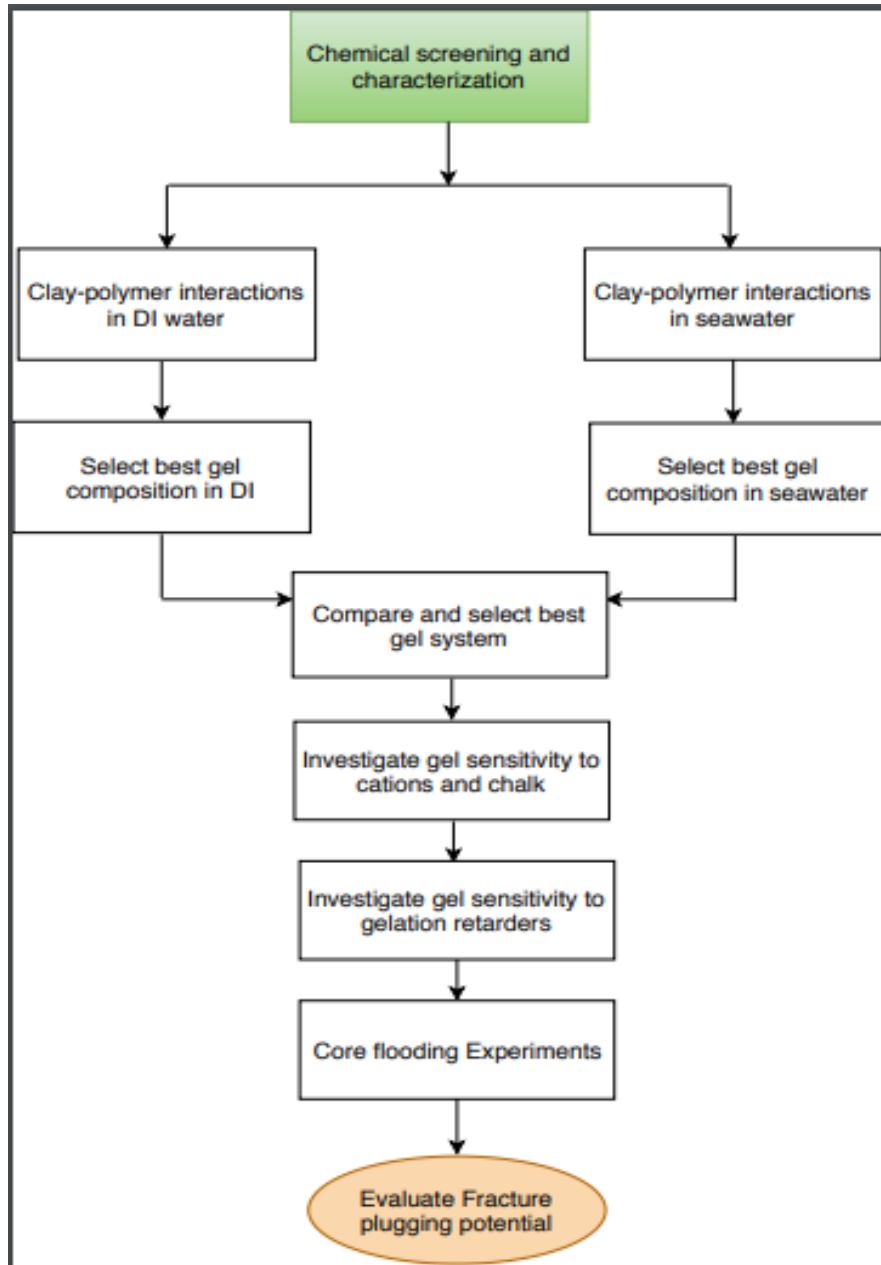
Figure 4.1: A schematic diagram of experimental scope

4.1   Chemicals

4.1.1   Polymers

Table 4.1 below presents the polymers used in the experimental work conducted in this thesis

Table 4.1: polymer description

Polymer	Molecular Weight	Supplier
HPAM (Flopam 3630)	18,000,000	SNF S.A.S
HPAM (DP/ERD 2161)	500,000	SNF S.A.S
Polyethylene glycol (PEG)	300,000	Sigma Aldrich
Gellan gum	500,000	Alfa Aesar

Hydrolysed polyacrylamide (HPAM) was selected for investigation in this research because of its popular use in polymer gels (Sydansk & Romero-Zeron, 2011). Two types of HPAM was used in this study; one with a high-molecular weight of 18,000,000 Daltons and the other with low-molecular weight of 500,000 Daltons). The need to maximize the injectivity of the gelant and achieve a low viscous gelant before injection led to the switch to a polymer with lower molecular weight. PEG is a water-soluble polymer with applications in industries in which polymer particle formation technology is very important, such as the pharmaceuticals, cosmetics, and food industries (Yoo et al., 2019). However, there has been some studies on its use in water conformance treatments, hence, it was selected for investigation in this research. Gellan gum is an anionic polysaccharide produced by the bacteria Sphingomonas elodea (Xu et al., 2019).

4.1.2   Clay

Table 4.2 presents the polymers used in the experimental work conducted in this thesis

Table 4.2: Description of Clays

Clay	Particle Size	Supplier
Laponite RD	25 nm	Alfa Aesar
Bentonite	2 μ or less	Ecca Holdings PTY

Laponite RD $Na^+_{0.7}$ [(Si_8 $Mg_{5.5}$ $Li_{0.3}$) $O_{20}(OH)_4$]$^{-0.7}$ is a synthetic layered silicate clay with an average lamellar structure diameter of 25nm, an average lamellar thickness of 1nm (Sidiq et al., 2009). Bentonite in sodium bentonite form is a colloidal forming clay used as a suspending agent in oil and gas applications.

4.1.3   Gelation Retarder

Various Low molecular weight glycol-based chemicals were added to laponite dispersions to investigate their potential for delaying gel formation. These chemicals are presented in table 4.3.

Table 4. 3: Gelation retarding chemicals

Chemical	Supplier
Polyethylene glycol (PEG)	SNF S.A.S
Butyl glycol	VWR international
Butyl diglycol	VWR international

4.2   Equipment/Materials

Experimental procedure and materials used are listed below:

1. Magnetic stirrer: Heidolph MR hei-standard stirrer was used for mixing solutions
2. Vortex Shaker: For mixing test tube gel solutions and evaluate gel strength
3. Rheometer: The Anton Paar MCR 301 rheometer was used for viscosity measurements
4. Oven: For heating gelant solutions at 50°c
5. Weighting balance: A Mettler Toledo PB303 balance was used to measure all chemicals and solutions
6. Test tube/conical flask: For storing NC gels

4.3   Chemical Screening and Characterisation

Chemical screening involves the evaluation of the potential of various polymers and clays in preparing nanocomposite gels. Literature review on nanocomposite gels provides different methods of making nanocomposite gels, the preparatory method used for this study was that reported by (Mohammadi et al., 2015), the slight difference in both approaches is the use of a crosslinker which was excluded in this study. This study is split into three parts.

1. Clay-polymer interactions in de-ionized water are examined. This was done by examining laponite-polymer interactions and bentonite- polymer interactions. The results from both tests are then compared and, the most promising gel composition is selected.
2. Clay-polymer interactions in seawater are examined. This was done by examining laponite-polymer interactions and bentonite- polymer interactions. The results from both tests are then compared, and the most promising composition was selected.
3. In this last part, the best gel composition for both de-ionized water and seawater are then compared, and the best gel composition is selected

4.3.1   Chemical screening and characterisation in deionized water

This sub-section presents the examination of clay-polymer gelation property in deionized water. Deionized water used in this experiment was produced in the laboratory. First, laponite-

polymer interactions in deionized water are evaluated, then bentonite-polymer interactions are also examined. Results from both tests are then compared.

4.3.1.1 Examination of Laponite-polymer gelation property

This section presents the examination of laponite clay interaction with the various polymers examined in this study. The experimental procedure is described below.

- Polymer solutions:

Bulk polymer solutions were prepared and then diluted to desired concentrations. Bulk polymer solutions of 0.06 wt% and 0.6 wt% were prepared by adding powdered polymer to deionized water. For example, when preparing the lower concentration (0.06 wt%), 0.06g of polymer was added to 99.94g of deionized water while mixing with a magnetic stirrer for 2hours. Conversely for the higher concentration 0.6 g of polymer was added to 99.4g of deionized water while mixing with a magnetic stirrer for 2hours.

- Laponite dispersions:

Various concentrations of laponite dispersions were investigated. However, the method of preparation remained the same, method of preparation is described as follows: (a) desired amount of clay was measured,(b) clay was added into already measured amount of deionized water while mixing with magnetic stirrer, (c) Dispersion was mixed until a clear homogenous dispersion was obtained. It is important to mention that due to the aging of laponite dispersions, the dispersions are prepared a few minutes before their use

- Nanocomposite gel preparation:

The steps followed for preparing nanocomposite gels are described as follows: (a) desired quantity of polymer was measured and placed into a test tube, (b) desired quantity of clay solution was then measured and added to polymer solution from 'a', (c) solution from 'b' was then mixed with a vortex mixer at speed 7 for about 1min.

- Gel characterisation:

Screening and characterisation of prepared nanocomposite gels were carried out by simple inversion or/and shaking of the tubes with a vortex mixer at speed 1 for 30secs and then visually inspecting gel status. Gels are then characterized based on the gel code described in table 5.1.

4.3.1.2   Examination of Bentonite-polymer gelation property

This section presents the examination of Bentonite clay interaction with the various polymers examined in this study. The experimental procedure for preparing and characterising the bentonite-based gels are same as that described for laponite-based gels.

4.3.2 Chemical screening and characterisation in seawater

This presents the examination of various clay-polymer nanocomposite gels in synthetic seawater. First laponite-polymer interactions in seawater was examined, then bentonite-polymer interactions were also examined. Results from both tests are then examined and compared.

4.3.2.1   Seawater

Table 4.4 below presents the composition of synthetic seawater used in the preliminary testing of polymer and clay gelation interactions. Seawater with composition above was prepared by adding the salts in their respective amount into 1litre of deionized water and a magnetic stirrer was used to mix the solution for 24hours.

Table 4. 4: Composition of prepared seawater

Salt	Chemical Formula	Concentration of salt in SSW (g/l)	Supplier
Sodium Chloride	$NaCl$	23.38	Merck Chemicals
Sodium Sulfate	Na_2SO_4	3.41	Merck Chemicals
Sodium Carbonate	$NaHCO_3$	0.17	Merck Chemicals
Potassium Chloride	KCl	0.75	Merck Chemicals
Magnesium Chloride hexahydrate	$MgCl*2H_2O$	9.05	Sigma-Aldrich
Calcium Chloride	$CaCl_2*2H_2O$	1.91	VWR international

4.3.2.2　　　　Examination of Laponite-polymer gelation property

This section presents the examination of laponite interaction with the various polymers in seawater. The experimental procedure is the same as that described for laponite-polymer interaction in deionized water except that deionized water was substituted for seawater.

4.3.2.3　　　　Examination of Bentonite-polymer gelation property

This section presents the examination of bentonite interaction with the various polymers in seawater. The experimental procedure is the same as that described for bentonite-polymer interaction in deionized water except that deionized water was substituted for seawater.
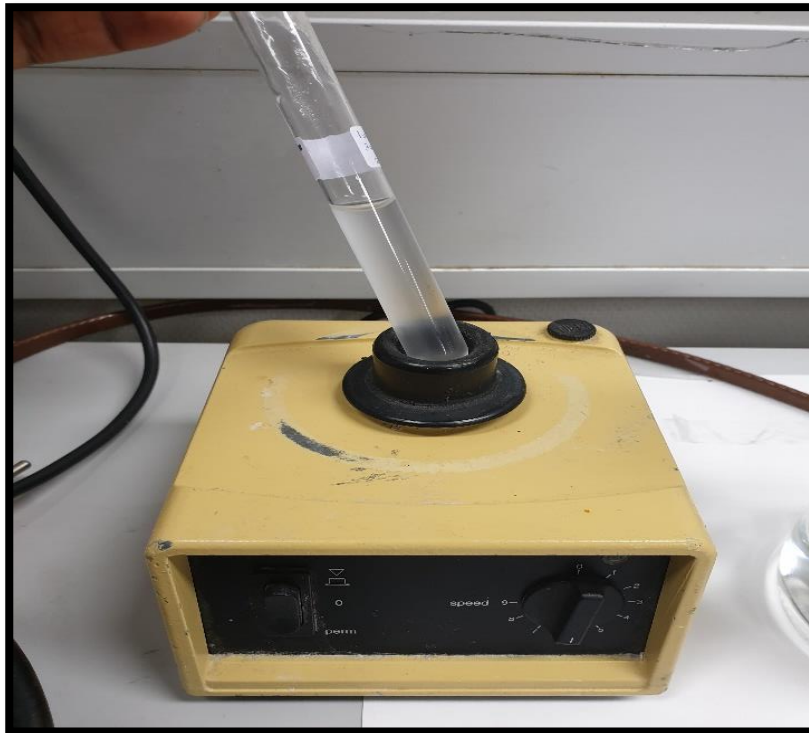

Figure 4.2: Mixing of polymer-clay solution with a vortex mixer

4.4 Investigation of clay sensitivity to Cations and Chalk

In-order to investigate the selected clay system to cations. Calcium chloride, potassium chloride, tap water, and chalk of different concentrations were added to pre-prepared clay solutions.

- **Preparation of salt solutions:**

Various concentrations of salt and chalk were tested. Bulk salt and chalk solutions were prepared and then diluted to desired concentrations. To prepare salt solutions, the following procedures were followed: (a) measured desired quantity of deionized water in a beaker, (b) measure desired quantity of salt and add to measured deionized water in 'a', (c) mix solution with a magnetic stirrer for 24hour to ensure a homogenous solution.

- **Preparation of chalk solutions:**

To prepare chalk solutions that would be used in monitoring the reaction of clay with chalk. The following procedure were followed: (a) a piece of chalk was crushed into powder (b) chalk powder was then weight in desired quantity and added to desired amount of deionized water (c) solution was then stirred with a magnetic stirrer and placed in the oven at 50°c for 1 day to enable dissolution of chalk particles (e) solution was then mixed with the magnetic stirrer again to ensure an homogenously dispersed solution.

- **Preparing clay-salt/ clay-chalk/ clay-Tap water solutions:**

To investigate clay sensitivity to salt/chalk solutions, the following procedures were followed: (a) measure desired quantity of clay dispersion in a test tube (b) add desired quantity of previously prepared salt/chalk (c) mix solution with a vortex mixer for 1min at speed 7 (d) tubes were then placed in the oven at 50°c and monitored over time. The procedure for preparing clay-tap water solution is slightly different. This time, the desired quantity of powdered clay was measured and added in tap water taken straight from the lab while mixing with a magnetic stirrer to ensure uniform dispersion of the clay

- **Gel Characterisation:**

Screening and characterisation of gels were carried out by simple inversion or/and shaking the tubes with a vortex mixer at speed 1 for 30secs and then visually inspecting gel status. Gels are then characterized based on the gel code described in table 5.1.

4.5 Effect of gelation retarders on Nanocomposite gels

Nanocomposite gels undergo a transitioning with time from a low viscosity solution to a rigid gel i.e. aging. The time required for the gelant solution to metamorphosize is known as the gel time. Nanocomposite gels are considered for use in-depth the reservoir and it is crucial to have proper understanding and control of the gelation process to ensure that gelation occurs in target areas at the right time and prevent injectivity problems. To achieve this, various low molecular weight glycol-based chemicals are investigated for their potential to retard gelation process.

- **Preparing clay-retarder solutions:**

Various types and ratios of clay-retarder solutions were prepared and investigated however the method of preparation remained the same and is described as follows: (a) measure desired quantity of deionized water (b)add desired quantity of retarder and stir with a magnetic stirrer for 5mins at 700rpm (c)add desired quantity of clay into the solution in 'b' (d) stir solution to ensure uniform dispersion of the clay (d) monitor gelant transition at room temperature and oven at 50°c. What is important to mention here is that only clay interaction with retarding agent is considered here because the aging characteristic of nanocomposite gels is a property of clay, hence it was not necessary to consider polymer.

- **Gel screening and characterisation:**

Gel was screened by monitoring the viscosity increase with time for all the systems, viscosity of the systems containing retarders were also compared with system without retarder. The retarder with the lowest viscosity evolution with time was then selected for core flooding experiment.

4.6   Fracture plugging potential of Nanocomposite gels

This section presents the investigation of the fracture plugging/ permeability reduction potential of nanocomposite gels. A schematic setup of the core flooding experiment is presented in fig 4.3 below. Three flooding experiments were carried out and are described in subsections below.

4.6.2   Preparation of Nanocomposite Gel system

The injected nanocomposite (NC) gelant system was prepared in the laboratory. It composed of 2.5wt% laponite, 2.5% polyethylene glycol (PEG), and 900ppm of the low molecular weight hydrolysed polymer acrylamide (HPAM). The preparatory steps of the gelant solution is described as follows: (a) measure desired quantity of deionized water in a flask (b) add desired
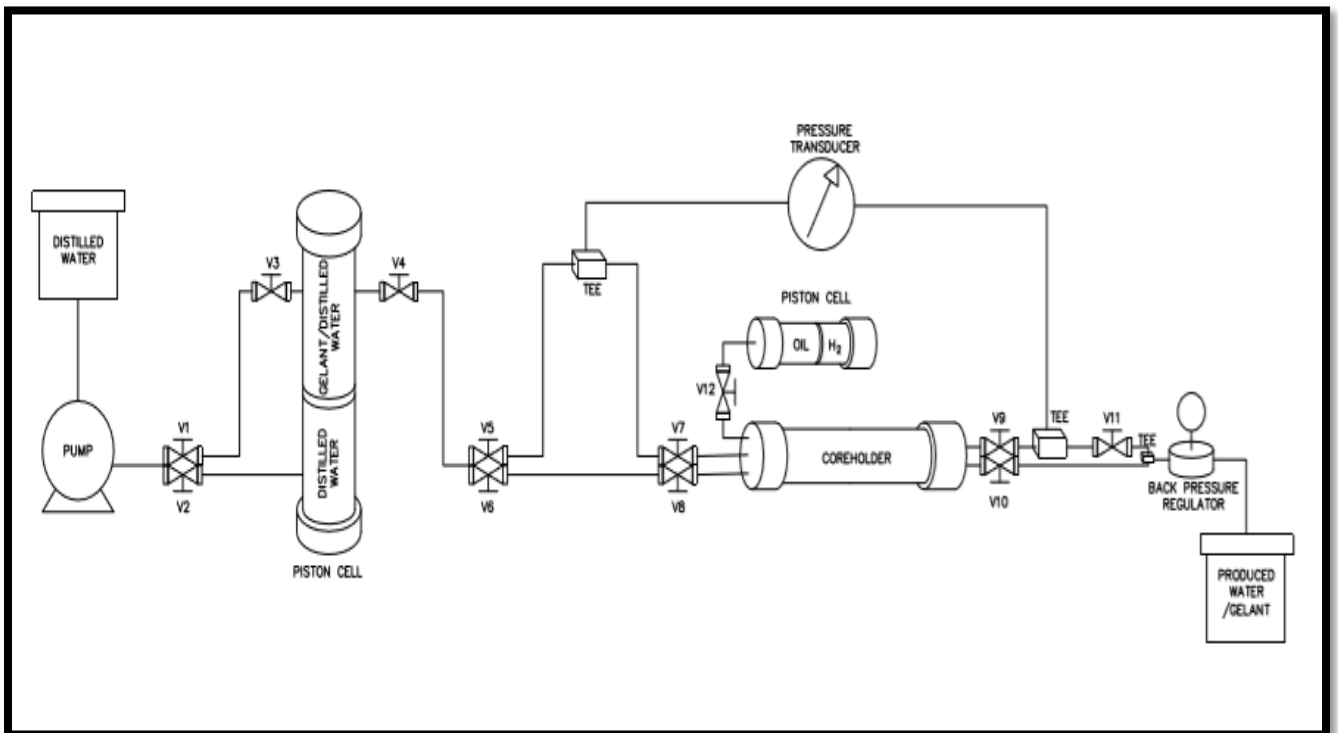

Figure 4.3: Schematic setup of experimental flooding process

quantity of PEG and stir with a magnetic stirrer for 5mins at 700rpm (c) add desired quantity of laponite into the solution in 'b' (d) stir solution to ensure uniform dispersion of the clay (d) in another flask, measured desired quantity of deionized (e) add desired quantity of HPAM and mixed with a magnetic stirrer (f) measured desired quantity of HPAM solution and add into pre-prepared solution in 'c' and mix with a magnetic stirrer. The prepared NC gelant was injected for experiment 1 and 2. However, experiment 3 involved the injection of laponite gel system. Its preparatory steps were the same as that described above except for that no polymer solution was added.

4.6.3 Experiment 1

Fracture model was made from a chalk core sample. Figure 4.4 presents the fracture model used in this experiment. The core sample has a diameter of 38.05mm and a length of 68.37mm. The fracture size is 68.37mm in length, fracture thickness was 1.25mm, and fracture breadth was 31.85mm. NC gel was injected, and the core flooding procedure was carried out at room temperature is described as follows:

- Core sample was mounted on a core holder and vacuumed off with a pump to ensure the removal of excess air before the core was then saturated with deionized water to calculate its pore volume and porosity. The pore volume is calculated by subtracting the wet weight of the core from the dry weight of core and the porosity is simply calculated using equation 4.1

$$\phi = \frac{Pore\ Volume}{Bulk\ Volume} \qquad\qquad 4.1$$

- Core was then injected with deionized water at room temperature using the flow rates at 0.1, 0.2, and 0.3ml/min until the differential pressure was stable. The corresponding pressure at each flow rate was then recorded. The absolute water permeability of core was calculated by the slope of the pressure gradient versus the rate, using Darcy's equation.

- To create a fracture in the core, the core was demounted, sliced into two halves and packed with a 1.25mm thick plastic cable ties and wrapped in Teflon to create a fracture model.
- The core was re-mounted and injected with deionized water to measure permeability after fracturing
- Prepared gelant was then injected into the core at a flow rate of 0.3ml/min, gelant was injected until gelant was produced.
- After the injection of the gelant, the core holder was disconnected from the set up and placed into the oven at 50°C for 3days (gel time from tube test = 1day, three days was selected to allow more time for gelant to react with chalk to activate gel formation) .
- Finally, the core holder was subsequently reconnected and deionized water was then re-injected at different rates until pressure differential was stable. The values were
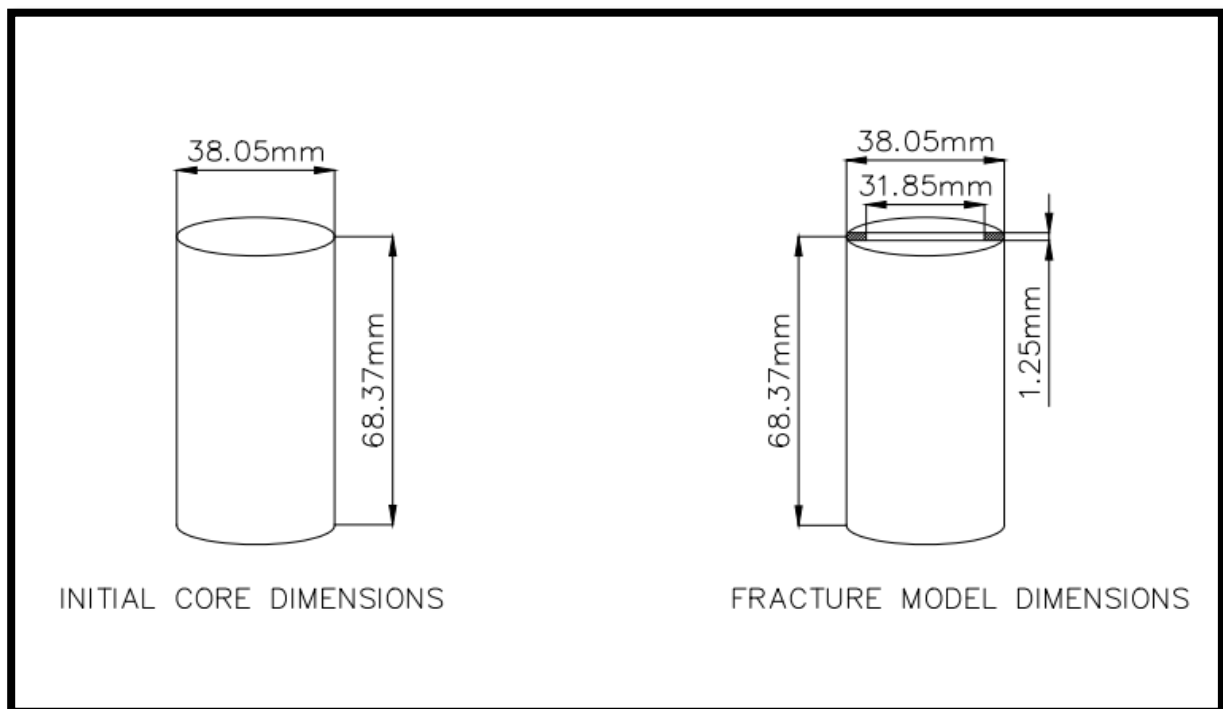

Figure 4.4: Fracture model description for core flooding experiment 1

recorded, and permeability of the core to water was calculated by the slope of the pressure gradient versus the rate, using Darcy's equation.

4.6.4 Experiment 2

Fracture model used in this experiment was also made from a chalk core sample. An illustration of this model is presented in figure 4.5. The fracture was packed with glass beads with the size range (43-52 μm). This fracture model was selected to create a more stable fracture and prevent fracture collapse. The core sample has a diameter of 38.05 mm and a length of 68.91 mm. The fracture size is 68.37 mm in length, fracture diameter is 4 mm. Prepared nanocomposite gel was injected into the core and the core flooding procedure was the same as that for Experiment 1. The only major difference was the use of a 3 mm thick slice of Bentheimer core with a permeability of around 2000 md as a filter on the outlet of the core to prevent the production of the glass beads.

Figure 4.5: Fracture model description for core flooding experiment 2

4.6.5   Experiment 3

For this experiment, pure laponite gelant solution with the same composition in selected NC gel was injected. The core type and fracture model remained the same as in experiment 2. The

fracture model is presented in figure 4.6. The fracture was packed with glass beads with the size range (43-52 μm). This fracture model was selected to create a more stable fracture and prevent fracture collapse. The core sample has a diameter of 38.05 mm and a length of 68.91 mm. The fracture size is 68.37 mm in length, fracture diameter is 4 mm. The selected Nanocomposite gel was injected into the core and the core flooding procedure is the same as that for Experiment 1. The only major difference was the use of a 3 mm thick slice of Bentheimer core with a permeability of around 2000 md as a filter on the outlet of the core to prevent the production of the glass beads.
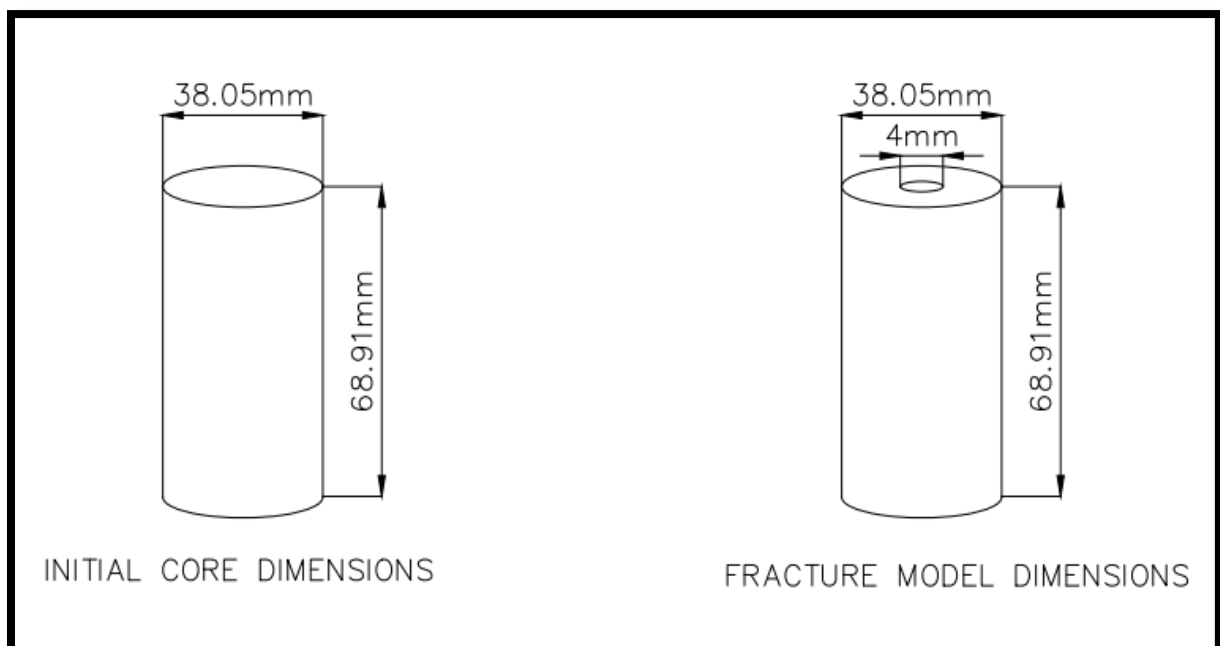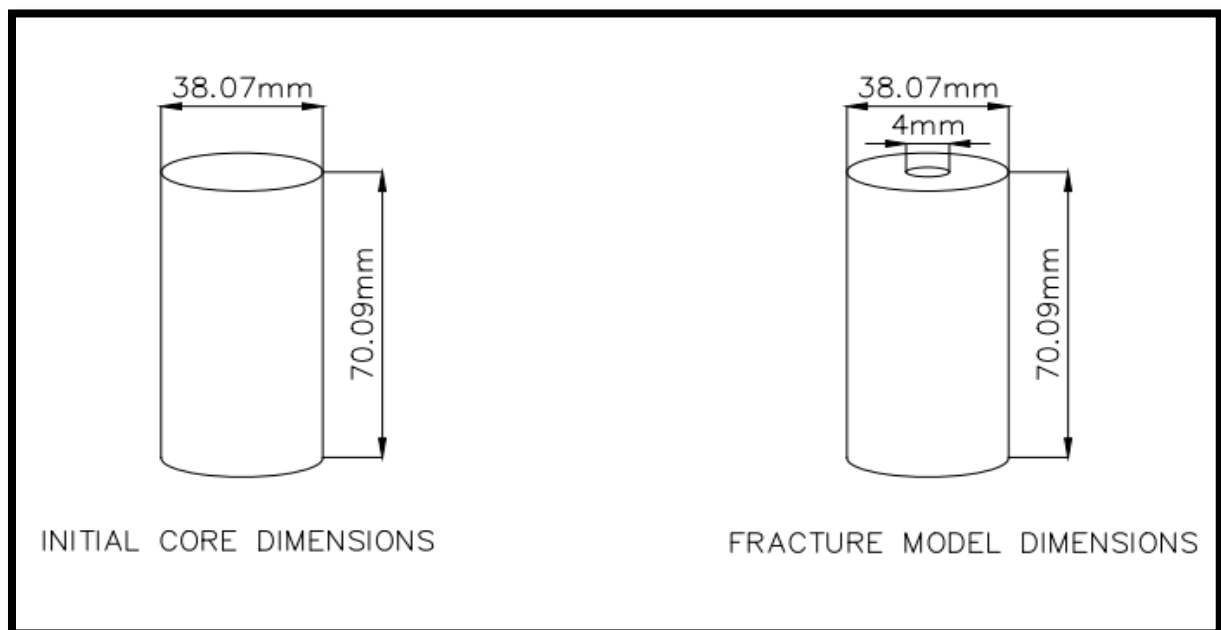

Figure 4.6: Fracture model description for core flooding experiment 3

Chapter 5   Results and Discussion

This chapter presents the results acquired during experiments. This chapter is divided in four section. Results, observations and discussions for each experiment is presented in each section. In order to properly monitor the gelation mechanism and describe the characteristics of the gels formed. A gel code based on (Skrettingland et al., 2012) is provided below in table 5.1

Table 5. 1 Gel classification and characterisation

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)

5.1   Chemical Screening and characterisation

Clay suspensions have a wide range of uses from cosmetics, agriculture, and even oil field applications. The behaviour of clay-polymer solutions can vary from that of either pure clay or pure polymer. Clay-polymer solutions contain special polymer-clay network in which polymer chains link clay particles and cause the formation of a gel structure.

This section presents the results of interactions between the various polymers and clay tested. First the results of laponite based nanocomposite gels prepared in deionized water are presented, thereafter the results for bentonite-based nanocomposite gels prepared in deionized water are presented. Thirdly, we compare the behaviour of both gels in deionized water

5.1.1 Characterisation of laponite gels in Deionized water

Upon the preparation of gelant solutions with the selected types of clay and polymers at different concentrations, different types of behaviours are observed. Table 5.2 presents the results for the various laponite and clay gel combinations investigated, and their respective concentrations.

Table 5.2: Preliminary investigation of Laponite-polymer interaction in deionized water, heated at 50°C for 1 day

Clay	Concentration (wt.%)	Polymer	Concentration (wt.%)	Gel code
Laponite RD	0.2	Gellan gum	0.03	1
Laponite RD	0.2	HPAM	0.03	1
Laponite RD	0.2	PEG	0.03	1
Laponite RD	1.0	Gellan gum	0.15	1
Laponite RD	1.0	HPAM	0.15	1
Laponite RD	1.0	PEG	0.15	5
Laponite RD	1.0	Gellan gum	0.3	2
Laponite RD	1.0	HPAM	0.3	3
Laponite RD	1.5	Gellan gum	0.3	4
Laponite RD	1.5	HPAM	0.3	5

Table 5.2 shows that at the lowest of concentration 0.2 wt.% laponite and 0.03 wt.% polymer, laponite based gels did not show any increase in viscosity regardless of the type of polymer used (HPAM, gellan gum and PEG). This behaviour is believed to be as a result of the low concentrations tested, which prevented the initiation of interaction between the nano-laponite

clay sheets and any of the polymers. Upon testing a slightly higher concentration laponite (1 wt.%) and polymer (0.15 wt. %), there was no significant improvement in viscosity of the gels formed for both laponite-HPAM and laponite-gellan gum solution.

However, an exciting behaviour was noticed with laponite-PEG gel, upon adding PEG solution to laponite dispersion and mixing with the vortex mixer, the low viscosity laponite-PEG gelant transforms into a deformable gel with high flow resistance i.e. This means that the gelant exhibits shear thickening behaviour which enables its transition from a low viscosity sol to a strong gel. This observation shares similarities with the experimental findings of Fall & Bonn (2012) where they reported shear thickening behaviour of laponite-PEG suspensions. They describe this process as a complex process involving the competing interactions between clay-clay particles as well as clay-polymer particles. When laponite clay is dispersed in water, the nano-sized clay particles become hydrated and electrostatic attraction between the negatively charged faces and positively charged edges of the clay discs causes the creation of a 'house of cards' structure which results in the formation of a gel structure (figure 5.1). Since laponite, particles when dissolved in water, form aggregates due to the electrostatic attraction between the negatively charged clay surface and its positively charged edges, a large number of aggregates implies lower clay volume available for interaction with polymer chains which then prevents the formation of a gel. However, the introduction of shear breaks up these large clay aggregates resulting in larger volumes of clay that can be bound by polymer chains resulting in the formation of a polyethylene glycol-laponite network (Fall & Bonn, 2012) which initiates the formation of a gel structure.
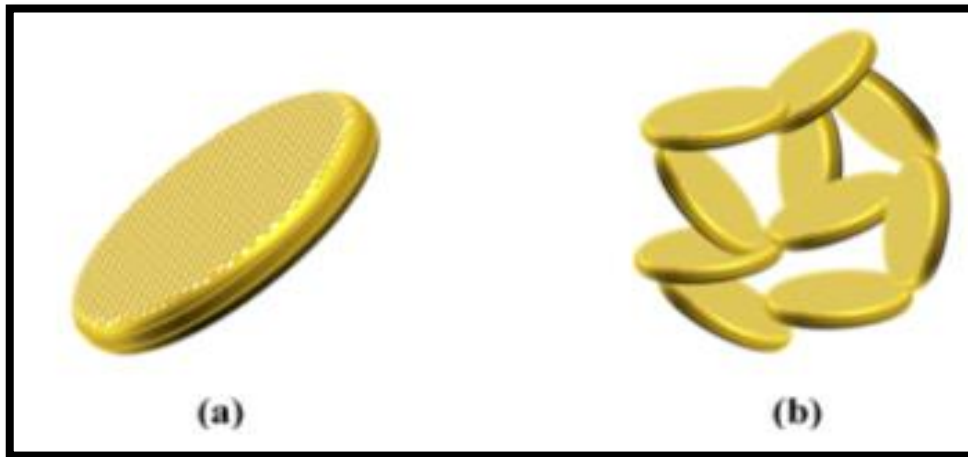
Figure 5.1 : (a) single laponite clay sheet, (b) house of cards structure (Bai et al., 2018)

A further increase in polymer concentration at 0.3 wt.% and laponite concentration at 1 wt.% produces a slight increase in original viscosity for laponite-gellan gum solution and a highly viscous and flowing gel for the laponite-HPAM solution. At the highest concentration of laponite tested (1.5 wt.%), the laponite-gellan gum solution produces a flow resistant gel with a deformable upper part while the laponite-HPAM solution produces a rigid gel that is resistant to flow upon inversion and shaking with the vortex mixer.

5.1.1.1   Discussion on Laponite based Nanocomposite gels in Deionized water

Figure 5.2 provides the comparison between the various laponite-polymer NC gels at various concentrations. No further test was carried out for higher concentrations of laponite-PEG based gels because of problems with its application in this research. Since the goal of this research was to form a gelant solution which should be easily injectable before it transitions into a rigid gel in the reservoir, the shear induced gelation property of laponite-PEG solutions will restrict their application for this purpose.

The presence of a significant gel structure was observed for higher concentrations of laponite-HPAM and laponite-gellan gum solutions (> 1wt.% laponite, 0.3 wt.% polymer). The strength of the gels formed also appeared to increase with increasing concentration of the laponite.

Laponite-HPAM solutions were also observed to form stronger gels when compared to Laponite-gellan gum solution. The intercalation of the polymer chains and clay particles to form a rigid gel structure confirm the advantageous effect of the nano-clay and polymer network present in NC gels (Yuan et al., 2014; Zolfaghari et al., 2006). Yuan et al., (2014) in their study, reported a tremendous increase in tensile strength of the gels formed when adding laponite into cellulose solutions.
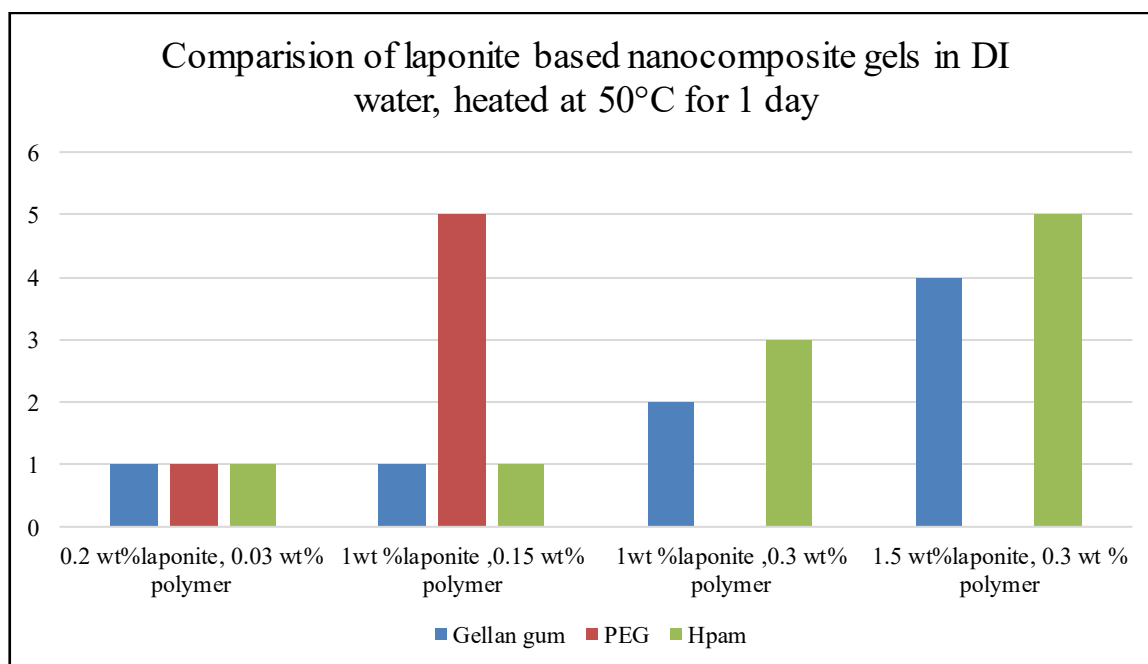

Figure 5.2: Comparison of laponite based Nanocomposite gels in DI water

5.1.2 Chemical Screening and characterisation of Bentonite gels in Deionized water

Table 5.3 presents the results for the various bentonite and polymer combinations investigated, and their respective concentrations.

Table 5.3: Preliminary investigation of bentonite-polymer interaction in deionized water, heated at 50°C for 1 day

Clay	Concentration (wt.%)	Polymer	Concentration (wt.%)	Gel code
Bentonite	0.2	Gellan gum	0.03	1
Bentonite	0.2	HPAM	0.03	1
Bentonite	0.2	PEG	0.03	1
Bentonite	1.0	Gellan gum	0.15	1
Bentonite	1.0	HPAM	0.15	1
Bentonite	1.0	PEG	0.15	1
Bentonite	1.0	Gellan gum	0.3	1
Bentonite	1.0	HPAM	0.3	1
Bentonite	1.0	PEG	0.3	1
Bentonite	1.5	Gellan gum	0.3	1
Bentonite	1.5	HPAM	0.3	1
Bentonite	1.5	PEG	0.3	1

It is seen from the table above that regardless of the concentration tested, bentonite solutions did not interact polymers tested to form gel. Bentonite based solutions resulted in the formation of a turbid solution which did not exhibit any viscosity increase when compared to its initial solution. Figure 5.3 shows a comparative plot of the various bentonite-polymer gels tested at various concentrations. Although, bentonite exhibits high swelling when it comes in contact with water, the bentonite clay particles could not be homogenously dispersed in the water and

seemed to settle at the bottom of the tube. The cause of this is suspected to be the larger size of the bentonite particles compared to laponite which prevented its interaction with the polymers.
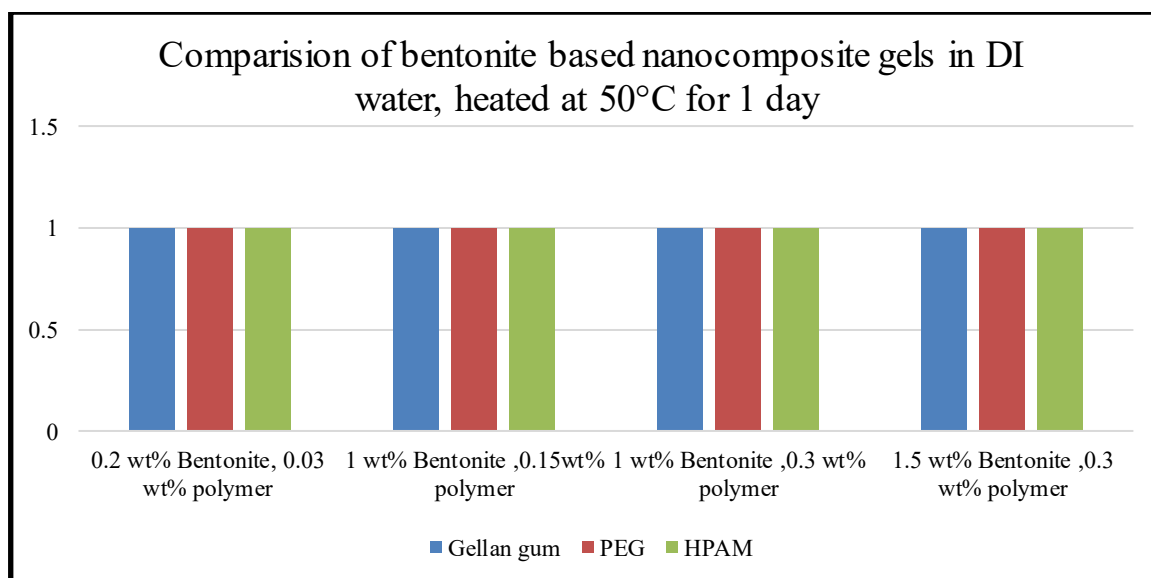

Figure 5.3: Comparison of Bentonite based Nanocomposite gels in DI water

5.1.3 Comparison Between Laponite And Bentonite Based Nanocomposite Gels prepared in Deionized water

Figure 5.4 presents the differences in performance between laponite and bentonite-based NC gels prepared in deionized water. For the lowest concentration at 0.2 wt.% Clay and 0.03 wt.% polymer, laponite based NC gels (presented on the left) behaved similarly to bentonite-based NC gels (presented on the right). They both have the gel code 1 which means that they did not show any increase in original viscosity, hence there was no formation of a gel structure.
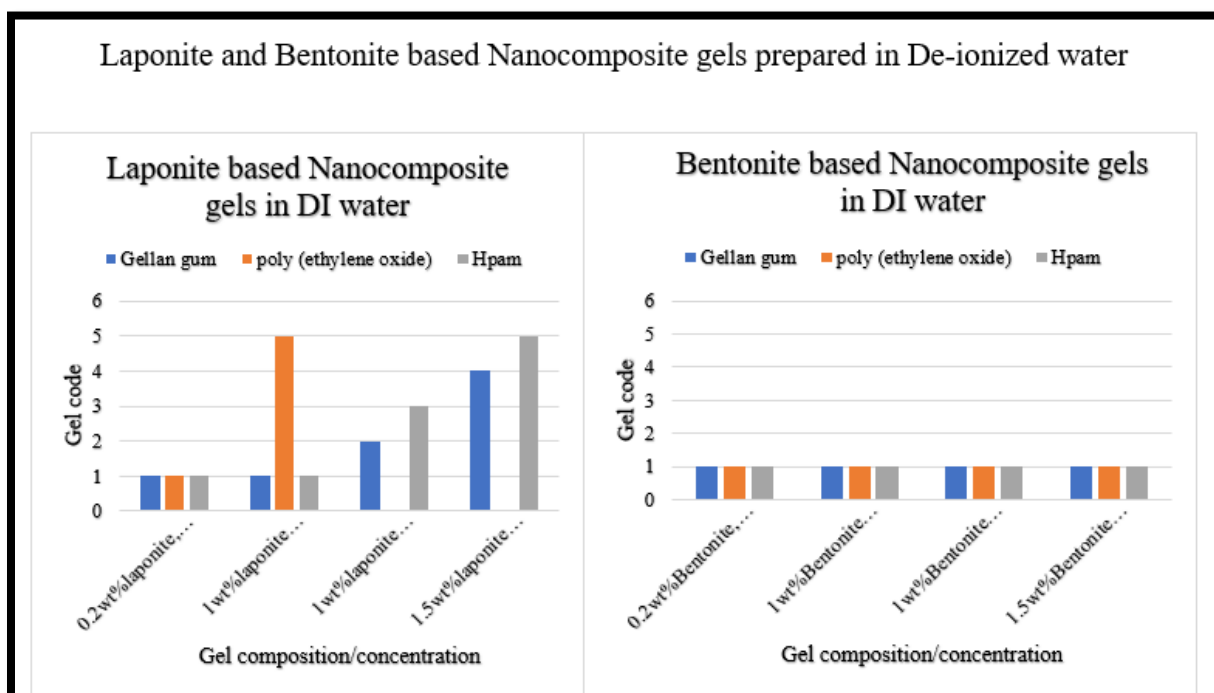
Figure 5.4: Laponite and Bentonite based nanocomposite gels prepared in deionized water

At 1 wt.% clay and 0.15 wt.% polymer, gellan gum and HPAM based solutions have the gel code 1 in both laponite and bentonite. However, laponite-PEG NC gel has the gel code 5, while bentonite- PEG NC gel has the gel code 1. This means that Laponite-PEG NC gels outperforms bentonite-PEG solutions by a factor of 5. The same trend is seen for all other higher concentrations where laponite based NC gels are seen to perform better than bentonite-based NC gels when prepared in deionized water. Based on this observation laponite was selected for further analysis.

5.1.4 Characterisation of Laponite gels in Seawater

Upon the preparation of NC gels with the selected types of clay and polymers at different concentrations, the characteristics observed are presented in Table 5.4

Table 5.4: Preliminary investigation of Laponite-polymer interaction in seawater heated at 50°C for 1 day

Clay	Concentration (wt.%)	Polymer	Concentration (wt.%)	Gel code
Laponite RD	0.2	Gellan gum	0.03	1
Laponite RD	0.2	HPAM	0.03	1
Laponite RD	0.2	PEG	0.03	1
Laponite RD	1.0	Gellan gum	0.15	1
Laponite RD	1.0	HPAM	0.15	1
Laponite RD	1.0	PEG	0.15	1
Laponite RD	1.0	Gellan gum	0.3	1
Laponite RD	1.0	HPAM	0.3	1
Laponite RD	1.0	PEG	0.3	1
Laponite RD	1.5	Gellan gum	0.3	1
Laponite RD	1.5	HPAM	0.3	1
Laponite RD	1.5	PEG	0.3	1

Regardless of the concentration tested, no gel structure was observed for all the concentrations of laponite and polymer solutions prepared in seawater (see figure 5.5). In fact, upon mixing powdered laponite with seawater, flocculation and sedimentation of the particles occurred, forming a white cloudy sediment at the bottom of the tube, an illustration of this is shown in figure 5.6. This observation shows slight similarities with the experimental findings of an older

study where they report similar flocculation of laponite in the presence of high NaCl concentrations (Mourchid et al., 1998). The flocculation of laponite in seawater is suspected to be as a result of the presence of high amount of cations in the seawater pre-mix, (BYK, 2016). Although there have been some studies in which laponite-based gel was prepared with seawater (Bai et al., 2018), the method of preparation involved adding powdered laponite clay into a solution of crosslinker and polymer prepared in seawater, while in this study, powdered laponite was added directly to seawater.
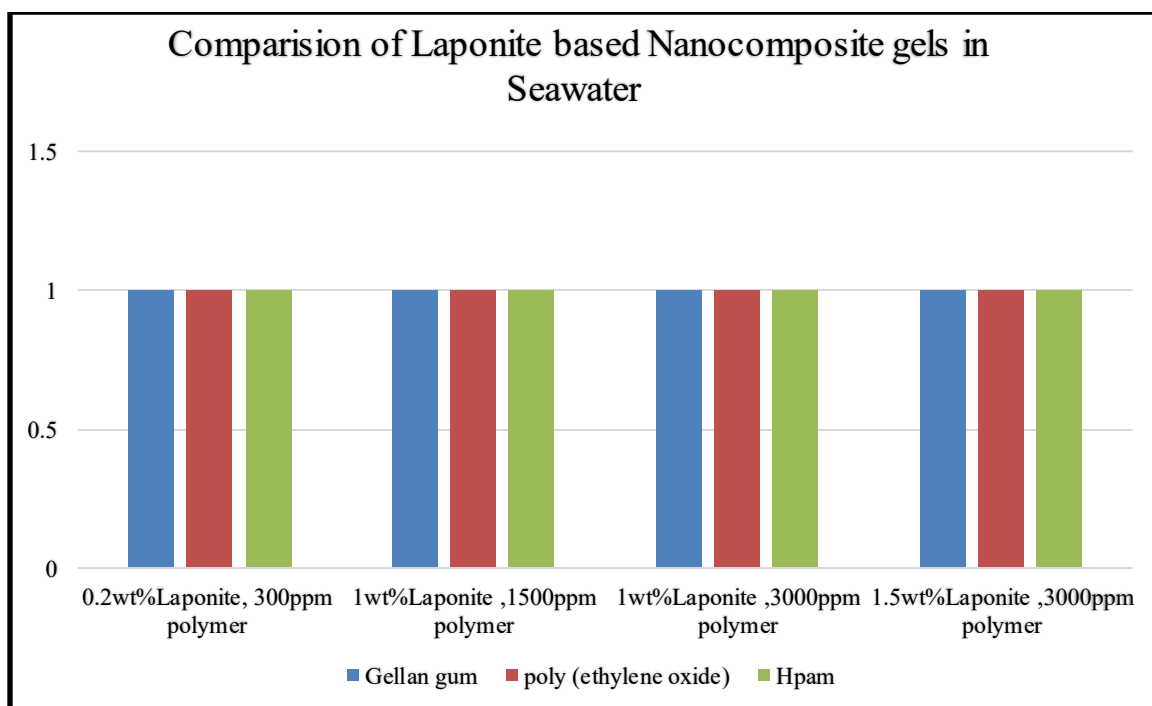

Figure 5.5: Comparison of laponite based nanocomposite gels in seawater

Figure 5.6: Flocculation of laponite when prepared in seawater

5.1.5   Chemical Screening and characterisation of Bentonite gels in Seawater

Table 5.5 presents the results for the various bentonite and polymer combinations investigated, and their respective concentrations

Table 5.5: Preliminary investigation of Bentonite-polymer interaction in seawater

Clay	Concentration (wt.%)	Polymer	Concentration (wt.%)	Gel code
Bentonite	0.2	Gellan gum	0.03	1
Bentonite	0.2	HPAM	0.03	1
Bentonite	0.2	PEG	0.03	1
Bentonite	1.0	Gellan gum	0.15	1
Bentonite	1.0	HPAM	0.15	1
Bentonite	1.0	PEG	0.15	1
Bentonite	1.0	Gellan gum	0.3	1
Bentonite	1.0	HPAM	0.3	1
Bentonite	1.0	PEG	0.3	1
Bentonite	1.5	Gellan gum	0.3	1
Bentonite	1.5	HPAM	0.3	1
Bentonite	1.5	PEG	0.3	1

It is seen from the table above that regardless of the concentration tested, bentonite gels have the gel code 1 and their solutions did not form gels with any of the polymers tested. A picture of the solution formed upon mixing bentonite solutions with polymers is presented in figure 5.7, the settling of the bentonite particles observed for deionized water were also observed for sea water. Figure 5.8 below shows the comparison between the various bentonite-polymer gel tests at various concentrations. The characteristics of the solutions were same as that observed for bentonite in deionized water.

Figure 5.7: Bentonite-based NC gel prepared in seawater
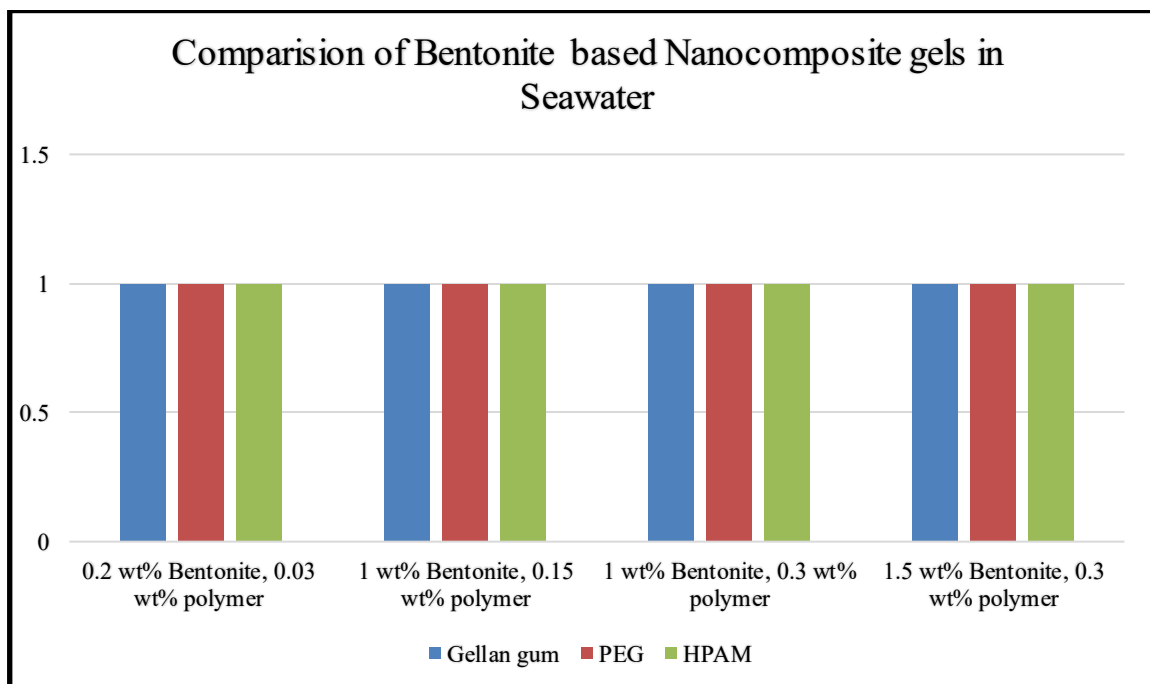
Figure 5.8: Comparison of bentonite-based nanocomposite gels in seawater

5.1.6 Comparison between Laponite and Bentonite Based Nanocomposite gels in seawater

Figure 5.9 below presents the comparison in performance between laponite and bentonite-based NC gels prepared in seawater. Both laponite and bentonite performed poorly and did not show any gel forming potential across all the concentrations tested. No distinction could be made on which was better between both gels

Comparison of laponite and bentonite-based nanocomposite gels prepared in seawater
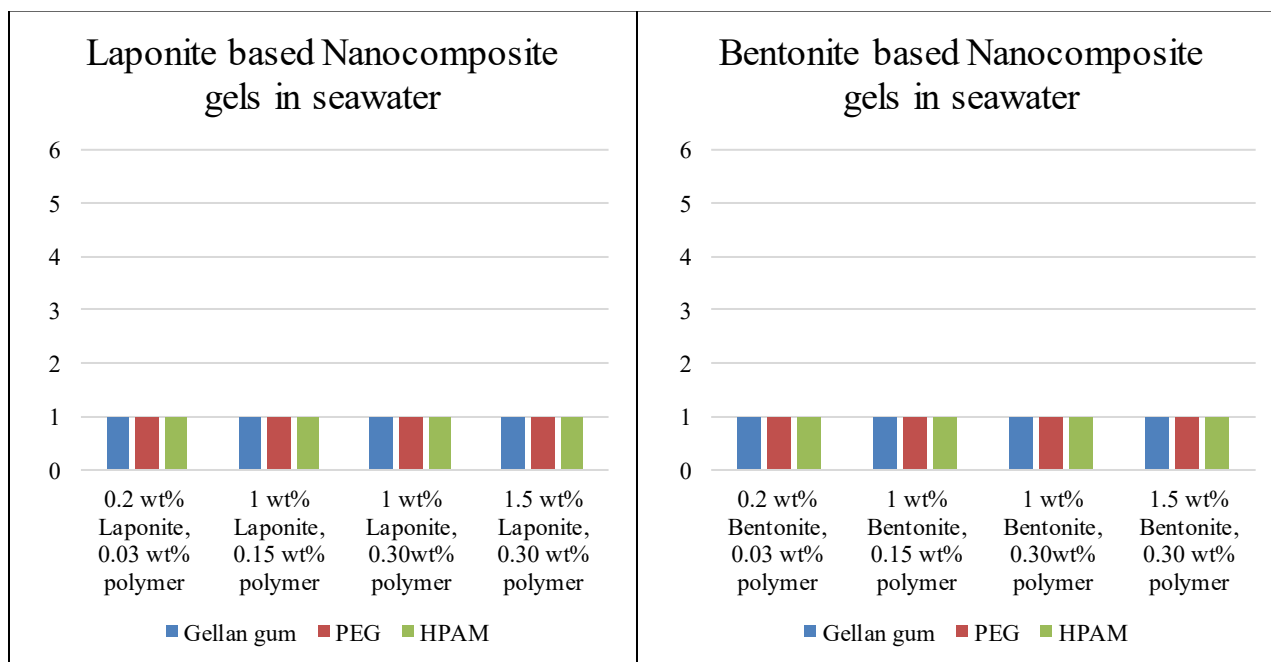
Figure 5.9: Comparison of laponite and bentonite-based nanocomposite gels prepared in seawater

5.1.7 Comparison Between Laponite Based Nanocomposite Gels prepared in Deionized and Seawater

In figure 5.10, the performance of the best performing gels in deionized and seawater are compared. Although both laponite and bentonite did not perform well in seawater, the performance of laponite-based gels in deionized and seawater were compared. It is clear that laponite performs better in deionized water compared to seawater at concentrations greater than 0.2 wt.%. For this reason, deionized water is chosen as the best solvent for preparing laponite nanocomposite gels.
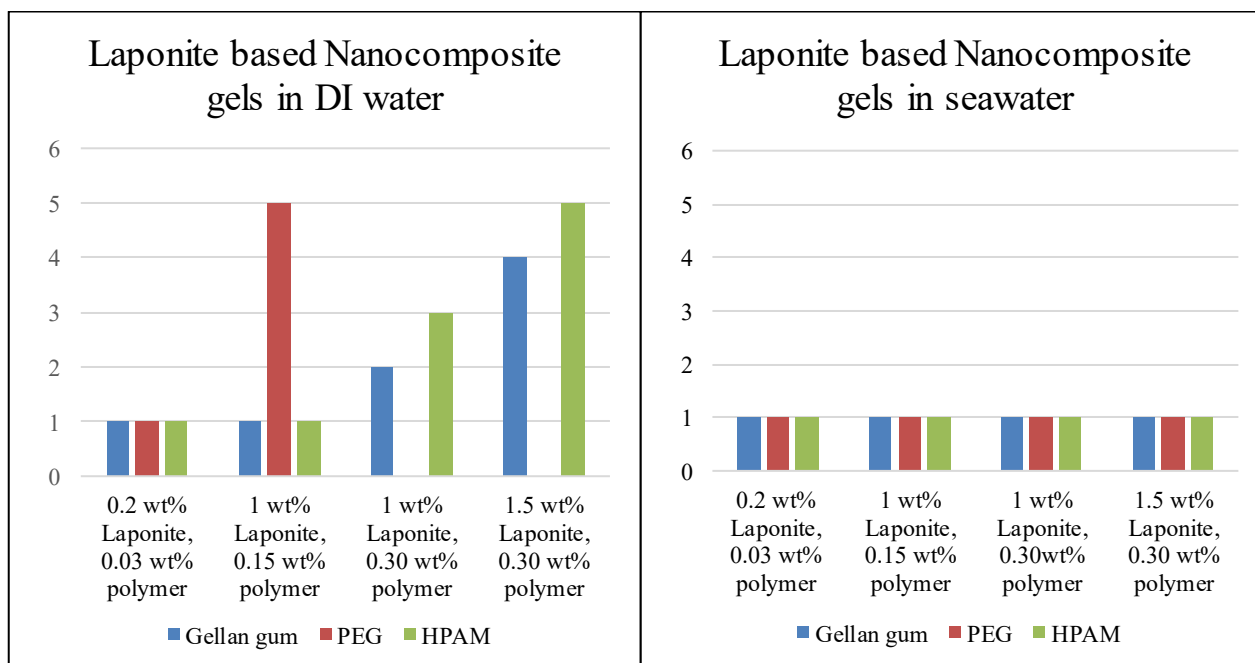
Figure 5.10: Performance of laponite in deionized versus seawater

5.2   Laponite sensitivity to Cations and Chalk

After the establishment of the gel forming potential of laponite gels in deionized water and their flocculation and sedimentation in seawater. It was decided to test the sensitivity of laponite to cations. The results after thermo ageing the gels for 1day at 50°C is presented below in table 5.6. It provides the comparison between laponite prepared in deionized water with no cations, Tap water, calcium chloride, potassium chloride and Chalk.

Table 5.6: Sensitivity of Laponite to Cations and Chalk

Clay	Concentration (wt.%)	Electrolyte	Concentration (mM)	Gel Code
Laponite RD	2	-	-	2
Laponite RD	2	Tap Water	-	4
Laponite RD	2	Calcium Chloride	3.23	3
Laponite RD	2	Calcium Chloride	6.46	4
Laponite RD	2	Calcium Chloride	12.92	5
Laponite RD	2	Potassium Chloride	3.23	3
Laponite RD	2	Potassium Chloride	6.46	4
Laponite RD	2	Potassium Chloride	12.92	5
Laponite RD	2	Chalk	3.23	3
Laponite RD	2	Chalk	6.46	4
Laponite RD	2	Chalk	12.92	5

At a concentration of 2 wt.% laponite forms a freely flowing viscous gel with a gel code 2 when prepared in deionized water. However, when prepared in Tap water (TW) at the same concentration, laponite forms a highly viscous flow resisting gel with gel code 4. When prepared with calcium chloride, laponite reacts with calcium to form a gel increasing in strength with increase in calcium chloride concentration. The same trend was also observed for potassium chloride with gel strength increasing with increase potassium chloride concentration. However, it was observed that potassium chloride induced gels showed shorter gel time than calcium chloride induced gels. Furthermore, chalk induced laponite dispersions were also observed to form gels similar to calcium chloride induced gels. Figure 5.11 presents a comparative plot of these results. A major inference from the results is that the presence of cations from salts and chalk can significantly speed up the laponite gelation process (Saha et al., 2015). This process is due to the screening of the electric double layer repulsion on clay sheets, leading to the aggregation of the clay particles and inherent formation of a gel structure. Laponite dispersions can exhibit a variety of physical characteristics from weak-sols to

viscoelastic gels or viscoelastic solids depending on laponite and salt concentration (Labanda & Llorens, 2008).
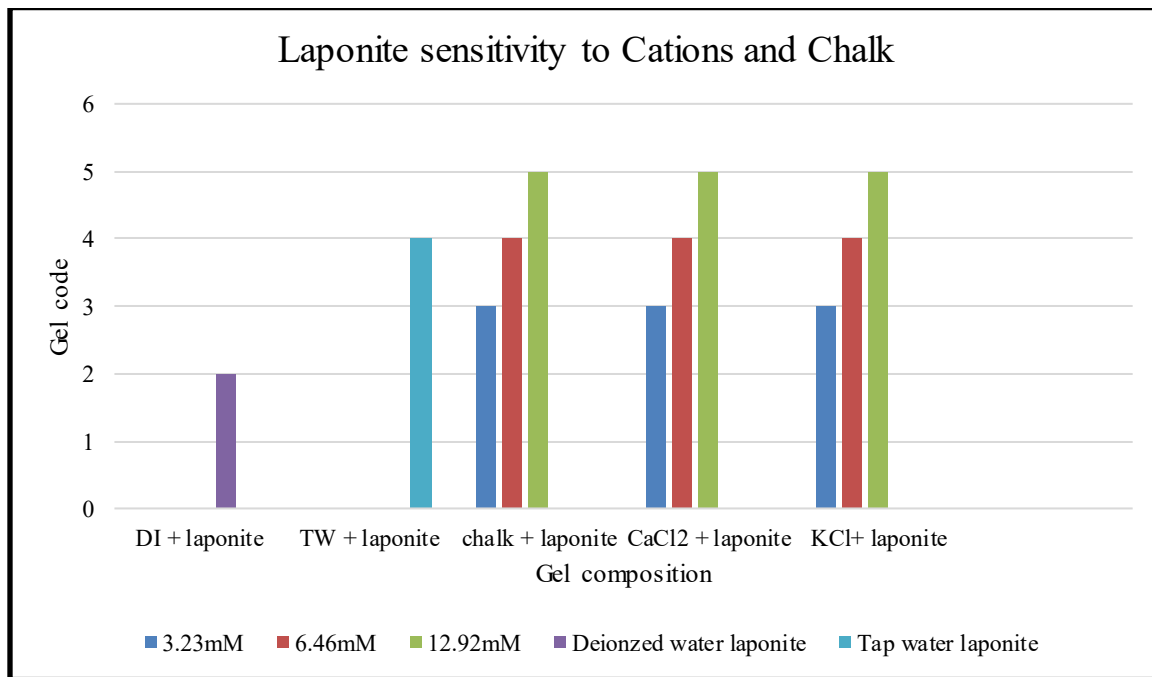
Figure 5.11: Laponite sensitivity to Cations and Chalk

5.3 Effect of Gelation Retarder

Various low molecular weight glycols were added to 4 wt.% laponite solution. This choice of concentration was chosen because laponite at this concentration forms a rigid gel in deionized water hence, it would be easier to see a definitive effect of the gelation retarders tested. First the clay and retarder are mixed in ratio 2:1 and 1:1 and allowed to age at room temperature for

1 day. Their behaviour was then analysed and presented in Table 5.7. It was observed that the retarders performed better when added to clay in ratio 1:1 i.e. when mixed in equal parts. This trend was same for all the retarders tested except for Butyl glycol which did exhibit any effect on laponite regardless of the ratio in which it was mixed. When butyl glycol was added to laponite, gelation occurred so fast that in occurred almost immediately the laponite clay was added. On the other hand, Butyl diglycol and PEG showed a significant effect on the

Table 5.7: Effect of gelation retarder				
Clay	Concentration (wt.%)	Retarder	Concentration (wt.%)	Gel Code
Laponite RD	4	-	-	5
Laponite RD	4	Butyl glycol	2	5
Laponite RD	4	Butyl glycol	4	5
Laponite RD	4	Butyl diglycol	2	4
Laponite RD	4	Butyl diglycol	4	3
Laponite RD	4	PEG (LMW)	2	3
Laponite RD	4	PEG (LMW)	4	2

physical characteristics of the gels formed. After 1 day at room temperature the gels remained as weak/flowing gels while the pure laponite solution formed rigid gel within 2hours of production. Upon comparison of the two promising retarders (BDG and PEG), PEG showed better performance because when mixed with laponite in ratio 1:1; a weaker gel with much lower viscosity is observed compared to the BDG induced gel. The Effect of the retarders on laponite viscosity few minutes after preparation when mixed in equal parts (1:1) at room temperature are presented in figure 5.12.
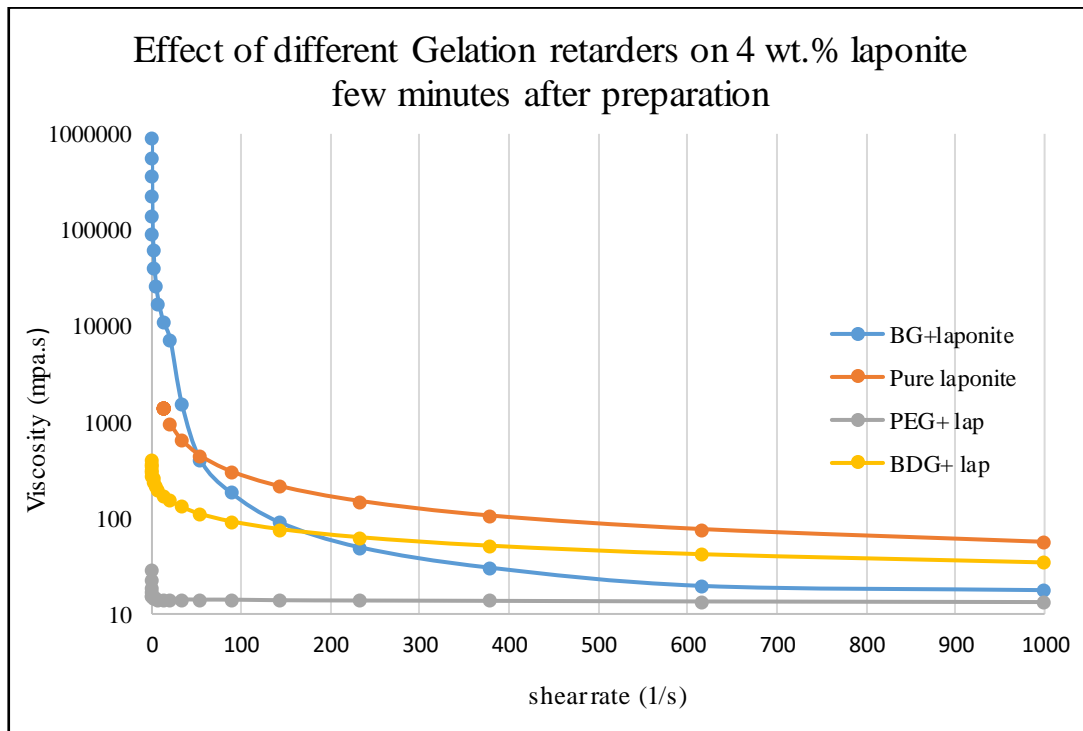
Figure 5. 12: Effect of different Gelation retarders on 4wt% laponite few minutes after preparation

From the figure above, PEG proves to be the most effective retarder tested as it shows around 100 times reduction in viscosity at low shear rates around 1 s^{-1} when compared to pure laponite. This magnitude of reduction in viscosity is seen to decrease to around 10 at higher shear rates. The same trend observed for PEG induced gels, although lower, is observed for BDG induced gels. The interaction of BG with laponite is quite different; very high viscosity around 1 million mpa.s is observed at lower shear rates around 1/s. However, the gel formed exhibit high thixotropic behaviour because its viscosity drops as low as around 20 mpa.s at higher shear rates around 700 s^{-1}. The addition of low molecular weight PEG and BDG to laponite dispersions can modify particle-particle interactions of the laponite particles (Labanda & Llorens, 2008; Mongondry et al., 2004). PEG can effectively screen inter-particle attraction of clay particles better than BDG by absorbing on the clay surface (steric repulsion). This effect is not only dependent on concentration but also molar mass of PEG (Mongondry et al., 2004). In the case of higher molecular weight PEG, the crosslinking of clay particles can occur resulting in the formation of a gel as observed in the previous experiments. This is the reason behind the choice of a lower molecular weight PEG for the purpose of this experiment. In

addition, a higher concentration of PEG implies that there are more polymer chains available to screen the clay particles. This explains why a weaker gel is formed upon the mixing of laponite/PEG in ratio 1:1

Since the main purpose of adding gelation retarders was to retard gelation until the gelant solution is injected in target zones, it is important that the gelation retarders would not impair the final strength of the gel formed. i.e., It was hypothesized that their effect would be overcome upon thermal aging of the laponite dispersion. To confirm this hypothesis, the retarder induced laponite solutions were placed in the oven at 50 ^{o}C and their transition was monitored over time. The results are the presented in figure 5.13. The plot of viscosity versus shear rate over time highlights the evolution of thermally aged laponite-PEG gels. A clear progressive increase in viscosity was observed as the laponite-PEG gel ages with time. The laponite-PEG dispersion transforms from a low viscous non-shear thinning solution into a viscous elastic almost solid gel after aging for 24hours at 50 ^{o}C.

The viscosity of the laponite-PEG gels at this time is slightly higher at lower shear rates than that of pure laponite. This implies that PEG only merely reduces the aggregation rate of the laponite particles (Mongondry et al., 2004). The electrostatic attraction of the laponite particles simply increases with the addition of temperature, overcoming the effect of PEG resulting in faster aggregation of the clay particles and accompanied increase in viscosity. Based on this observation, PEG was selected as a retarding agent for the concluding part of the experiments
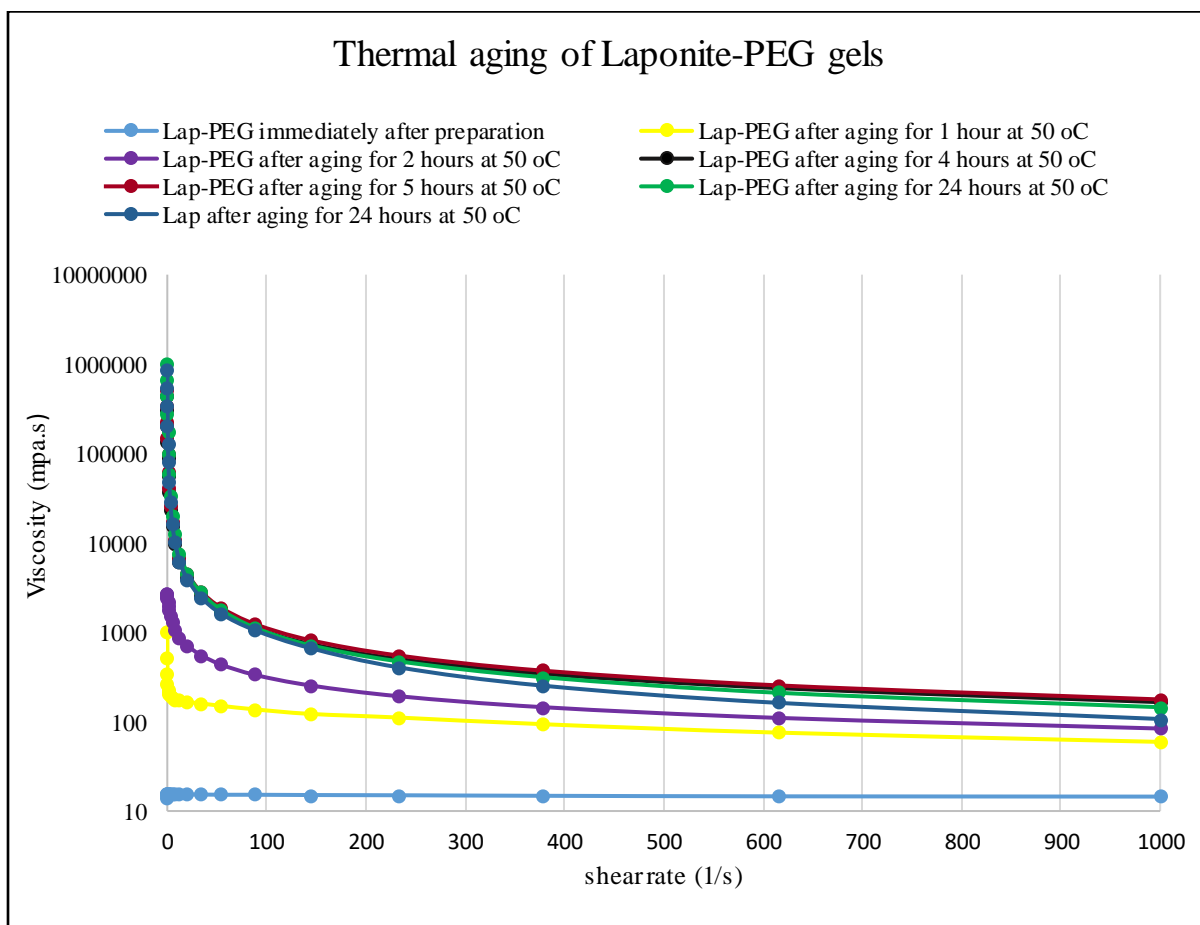
Figure 5.13: Thermal aging of laponite-PEG gels

5.4   Nanocomposite Gels as a Fracture Plugging Agent in chalk

In this section, the potential of nanocomposite gels as a fracture plugging agent in chalk was investigated. Nanocomposite gel composing of low molecular weight HPAM (DP/ERD 2161) with molecular weight of 500,000 Daltons, laponite and PEG was injected to fractured chalk. Its performance was then compared to that of pure laponite gel in chalk. It should be noted here that no salt/chalk was added into the gelant solution. This is because it is hypothesized that the injected gelant solution would react upon contact with the chalk at high temperature to form a rigid gel. The results of the experiments are presented in sections below.

5.4.1 Core flood 1

This section presents the results for the first core flooding experiment. The porosity of the chalk core sample was calculated to be 0.39. The fracture model created with cable ties and presented in figure 4.4 was used. The injected nanocomposite gel composition was 2.5wt.% laponite, 2.5wt.% polyethylene glycol (PEG), and 0.09 wt.% low molecular weight HPAM. This composition was same for core flood 2. The viscosity of the gelant solution is presented in figure 5.14.
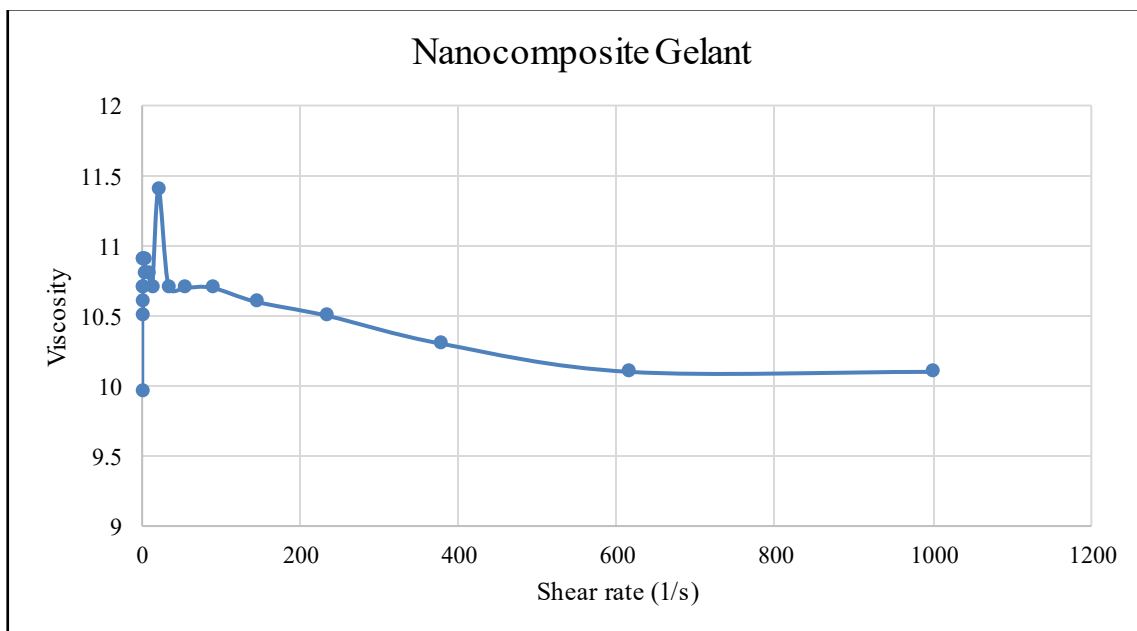
Figure 5.14: Viscosity versus shear rate of nanocomposite gelant (2.5wt.% laponite, 2.5wt.% PEG, and 0.09 wt.% HPAM)

The viscosity measurement of the NC gelant solution presented in figure 5.14, has been taken few minutes after preparation. The low viscosity and irregular shape of the curve at lower rates is as a result of the complex effects of the laponite-polymer, laponite-laponite and laponite-PEG particle interaction. The slight decrease in viscosity with shear is due to breaking of some of the formed laponite-laponite aggregates with increasing shear rate.

Table 5.8: The results of Core-flooding experiment 1

Before Fracture		After Fracture		After NC gel treatment	
Rate (ml/min)	Δp (mbar)	Rate (ml/min)	Δp (mbar)	Rate (ml/min)	Δp (mbar)
0.3	2900	3	15.0	0.3	760
0.2	1950	2	10.5	0.2	519
0.1	900	1	4.5	0.1	255
$k_w = 1.0$ md		$k_w = 1929$ md		$k_w = 3.5$ md	
		$F_{rrw} = 550$			

Before the fracturing of the core sample, the injection of water at the rates 0.1, 0.2, and 0.3 ml/min produced high differential pressures (900, 1950, 2900 mbar) respectively, increasing almost linearly. However, upon fracturing of the core sample, injected water seemed to simply channel through the created fractures resulting in the measurements of lower differential pressures and consequently higher permeabilities. The results of core-flooding test before and after treatment with NC gel, presented in table 5.8, showed that the studied NC gel formed a resistance to the flow of water, reducing water permeability from 1929 md to 3.5 md. The resistance factor for water was calculated to be around 550. This indicated the good performance of this gel in reducing water permeability.

However, during the run of this experiment, several problems were encountered. Upon the injection of water after the NC gel injection, the differential pressure rose quickly up to 5bar and the pressure of the pump also rose up to its pre-set safety pressure limit of 10bar. As a result, the pump was turned off. Due to this problem, face plugging of the core by the gel was suspected to have occurred and therefore the core was de-mounted and a thin slice of about 9mm was sliced off the inlet face of the core. The core was then remounted, the overburden pressure was increased to 20bar, and the pump safety limit was increased to 15bar. Water injection was then resumed to calculate water permeability after the NC gel treatment. Upon removal of the core after the experiment, it was observed that the cable ties which served as a bridge for the fracture had collapsed into the chalk. This caused the closing of the fracture aperture that was created earlier. This is shown in figure 5.15 below. Although upon closer

inspection of inside the fracture, gel was observed to have formed on the surface of the chalk. It was quite unclear as to whether the permeability reduction observed was mainly as a result of the gel, as a result of the collapse of the cable ties due to high overburden, or a combination of both effects.

Figure 5.15: collapsed fracture aperture, (a) before removing the core plug from the core sleeve, (b and c) upon removal and closer inspection of the core plug

5.4.2 Core flood 2

This section presents the results for the second core flooding experiment. The porosity of the core sample was also calculated to be 0.39. The fracture model 2, which was filled with glass beads presented in figure 4.5 was used in order to maintain a more stable fracture model. The

injected nanocomposite gel composition is same as that presented in core flood 1. The results of the core flood experiment are presented in table 5.9.

Table 5.9: Results of Core-flooding experiment 2

Before Fracture		After Fracture		After NC gel treatment	
Rate (ml/min)	Δp (mbar)	Rate (ml/min)	Δp (mbar)	Rate (ml/min)	Δp (mbar)
0.3	2900	3	270	0.3	2700
0.2	1930	2	165	0.2	2004
0.1	960	1	77	0.1	1125
$k_w = 1.1$ md		$k_w = 106$ md		$k_w = 1.6$ md	
		$F_{rrw} = 65.4$			

Before the fracturing of the core sample, the water injection at rates 0.1, 0.2, and 0.3 ml/min produced high differential pressures (960, 1930, 2900 mbar) respectively, increasing almost linearly just as observed in experiment 1. A permeability value of 1.1md was calculated for the measured rates and differential pressures using Darcy's law. However, upon fracturing of the core sample, injected water seemed to simply channel through the created fractures resulting in the measurements of lower differential pressures and consequently higher permeability (106 md). We observe a lower permeability value (106 md) compared to experiment 1 (1929 md) here after fracturing due to the different fracture pattern and aperture in both fracture models. After the NC gel treatment, subsequently injected water was met with high flow resistance leading to high differential pressure measurements. The results of core-flooding test before and after NC gel treatment, presented in Table 5.9 showed that the studied NC gel could reduce water permeability from 106 md to 1.6 md. The resistance factor for water due to NC gel treatment was calculated to be 65.4. This indicated the good performance of this gel in reducing water permeability.

During physical inspection of the core sample after the experiment (see figure 5.16), gel was observed to have set in the fracture forming a rigid barrier to the flow of water in the fracture

previously formed in the middle of the core, resulting in the diversion of subsequent injected water in the matrix. This same observation was reported by Bai et al., (2018) during their cubic fracture core model treatment with laponite/polymer dispersed gel.
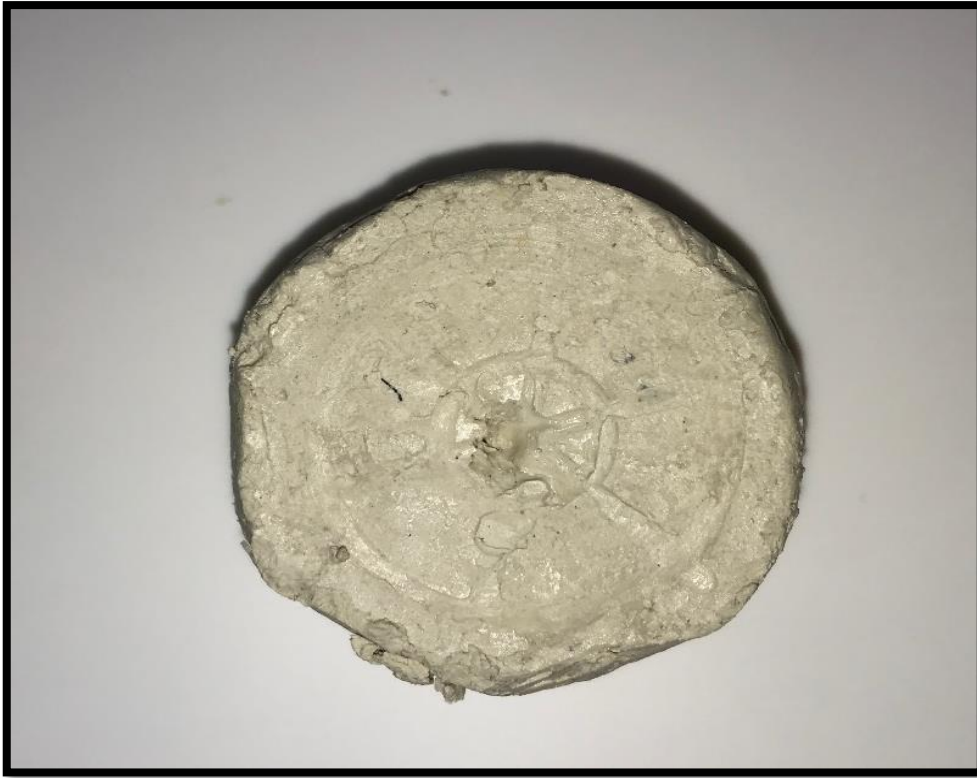

Figure 5.16 : Fracture plugging of Nanocomposite gels

5.4.3 Core flood 3

This section presents the results for the third core flooding experiment. The porosity of the core sample was also calculated to be 0.39. The fracture model 3, also filled with glass beads presented in figure 4.6 was used in order to maintain a more stable fracture model. Laponite gel with composition is 2.5 wt.% laponite and 2.5 wt.% PEG was injected. The viscosity of the gelant solution and results of the core flood results are presented in figure 5.17 and table 5.10 respectively.
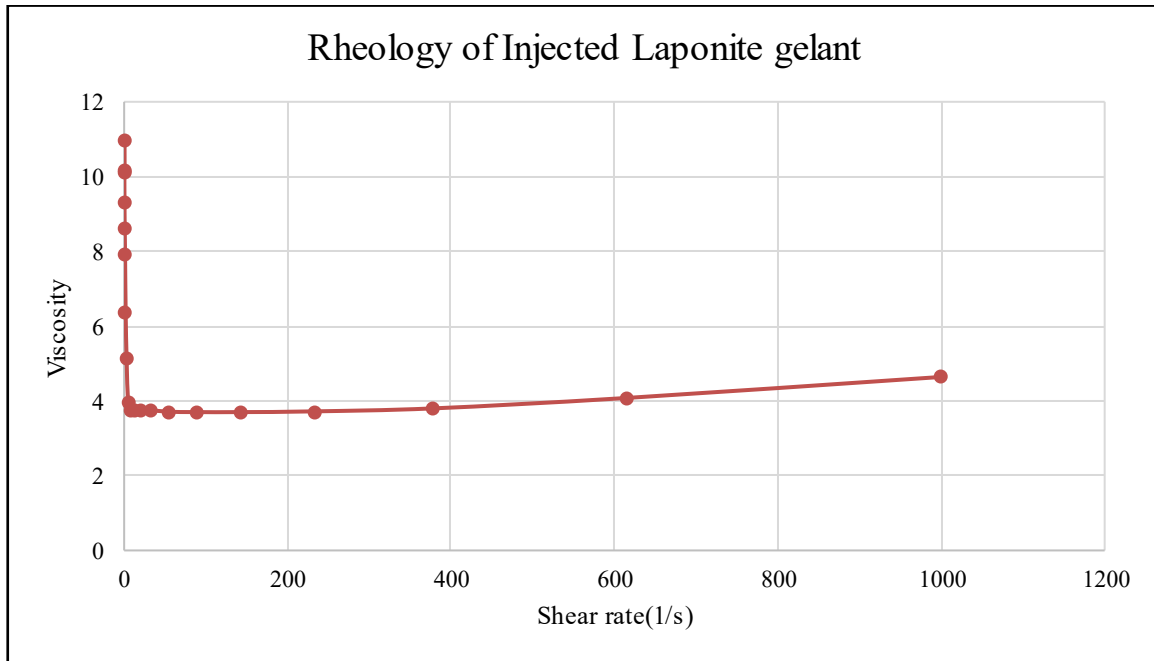
Figure 5.17: Rheology of Injected laponite gelant

The viscosity measurement of the injected laponite gelant presented above was taken few minutes after preparation. The sharp drop in viscosity is due to breaking of laponite-laponite aggregates as shear rate increases.

Table 5.10: Results of Core-flooding experiment 3

Before Fracture		After Fracture		After NC gel treatment	
Rate (ml/min)	Δp (mbar)	Rate (ml/min)	Δp (mbar)	Rate (ml/min)	Δp (mbar)
0.3	2890	3	1500	1	3325
0.2	1940	2	1132	0.7	2390
0.1	965	1	561	0.5	1750
$k_w = 1.1$ md		$k_w = 22.2$ md		$k_w = 3.8$ md	
		$F_{rrw} = 5.8$			

After the treatment of the core sample with laponite gel, the injection of water was also met with significant resistance just like in the case of NC gel. The differential pressures recorded during the injection of water were also high and the corresponding permeability value of 3.8 md was measured. The results of core-flooding test of laponite gel, presented in Table 5.10 showed that the studied laponite gel could reduce water permeability from 22.2 md to 3.8md. The resistance factor for water due to the treatment of the core sample was calculated to be 5.8. This suggest that laponite gels have a potential for plugging fracture and reducing the permeability of water in fractured core sample. During physical inspection of the core sample after the experiment, gel was also seen to have set in the fracture as observed for the core sample treated with NC gels in the previous section.

5.4.4 Discussion of Nanocomposite Gels versus Laponite gels

From the viscosity measurements presented in figure 5.18, nanocomposite gelant exhibited higher viscosity than laponite gelant due to the presence of polymer in their structure. This implies a favourable synergy between laponite and polymers similar as that reported by Mohammadi et al., (2015). The results from the core flooding test showed that both nanocomposite and laponite gels can form an effective barrier to the flow of water in fractures.
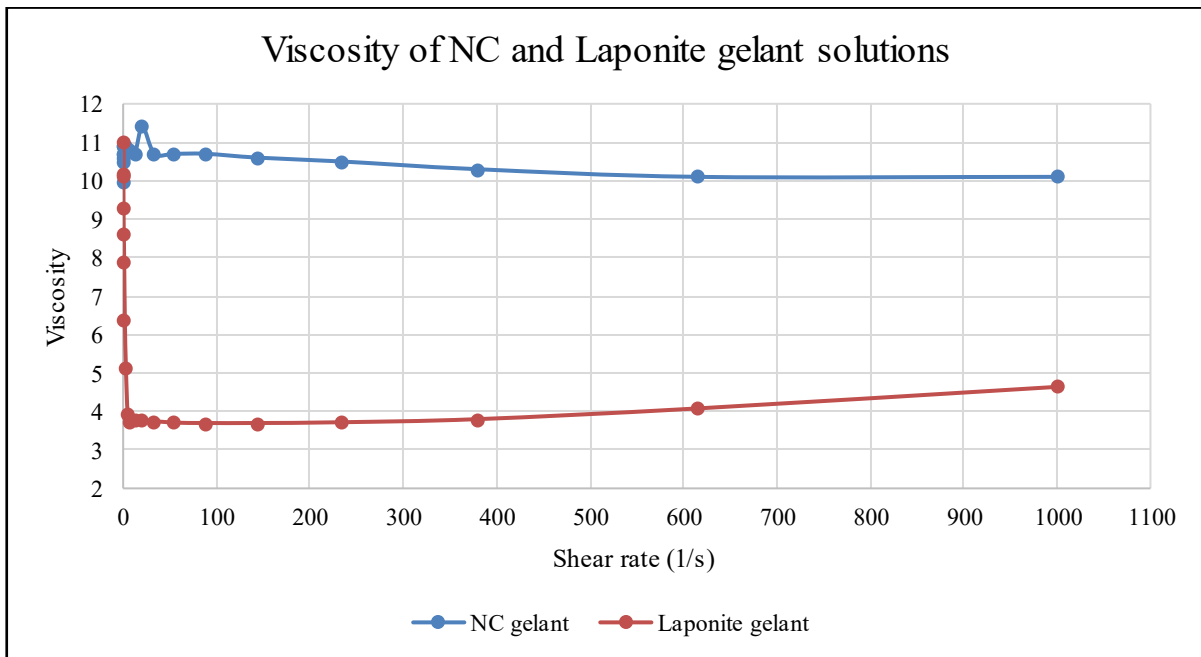
Figure 5.18: Rheological behaviour of NC and Laponite gelant

This is shown in the permeability reduction observed for both gels after injection. However, the difference in degree of packing of the glass beads in the fracture resulted in different permeability values after fracture preparation. The interaction between injected gelant and chalk in the core plug was also observed from the produced/ gel effluent (see figure 5.19). The produced gel effluent formed a cloudy rigid gel compared to the transparent/clear gels that are formed in the absence of chalk indicating the interaction of the injected gelant and the chalk in the fracture surface of the core plug. This confirms the hypothesis made earlier that injected gelant solution can react with chalk inside the core plug. The observation of the rigid gel formed in the fracture due to the treatment with NC gels presented in figure 5.16 indicates that nanocomposite gels can serve as an effective water/fracture plugging agent in chalk. This observation shows some similarities with that described by Bai et., al (2018).
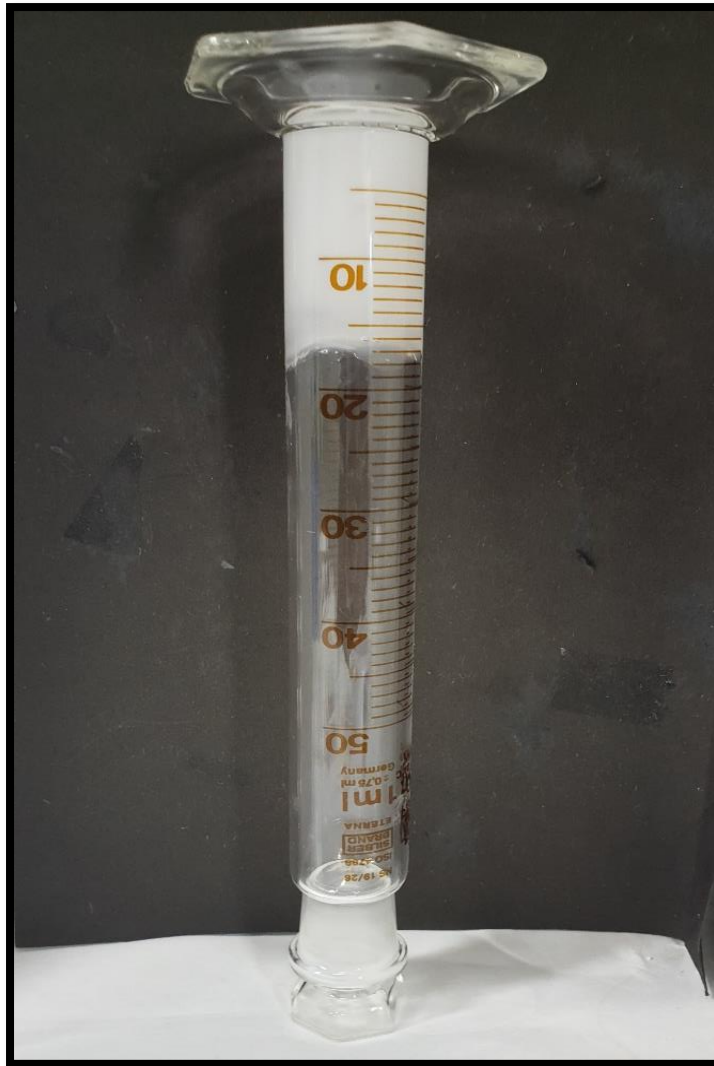
Figure 5.19: Gelant interaction with chalk in produced gel effluent

In their experimental investigation, they reported the selective filling of laponite/Polymer dispersed gels in fractures and also the subsequent plugging of fractures in a cubic fracture core model by the gel. However, there are slight differences in the composition of their gel and that used in this study, for example, their gelant solution composed of laponite, low molecular non-ionic polymer, a crosslinker N,N'-Methylene bisacrylamide and an accelerating agent ammonium persulfate. Despite the differences in composition of the gels the mechanism of the fracture plugging process in both tests are quite similar. Fracture plugging and corresponding reduction in permeability of water reported for both NC and laponite gels suggest the

effectiveness of both gels as a fracture plugging agent in Chalk. A higher residual resistance factor of 65.4 measured for NC gels compared to 5.8 for laponite gels suggests that NC gels provide better fracture plugging treatment. However, only a single experiment of both gels has been carried out, and at this time to the best of the author's knowledge similar studies with laponite gels has not been reported. It is therefore proposed that experimental investigations should be made to properly evaluate and compare the performance of both gels.

The core flooding tests and results reported in this thesis are not without limitations. In this thesis, reservoir conditions could not be fulfilled because gelant injections were carried out at room temperature, while gel activation was carried out in the oven at 50 ^{o}C. Inconsistent packing of the fracture with glass beads also prevented similar permeability measurements after fracturing the core plug. Regardless of these limitations, the permeability reduction of nanocomposite gels in chalks suggest them as an effective water/fracture plugging agent.

5.5   Recommendations for further work

The work done in this thesis has examined nanocomposite gels prepared with laponite and hydrolysed poly acrylamide (HPAM). Their potential as a fracture plugging agent in chalk has been confirmed. However, in-depth studies should be done to properly qualify the system. The following recommendations for future work can enhance our knowledge and effectively quality nanocomposite gels as water conformance agent in fractured chalks.

1.   X-ray diffraction analysis should be carried out to investigate the presence and degree of polymer-clay intercalation in prepared NC gels
2.   Additional characterisation of the NC gels could be done to determine monitor the transitional phases of the sol-gas transition and determine more accurately, gel times of the NC gels possibly in hours and minutes
3.   Experimental and rheological tests should be done to compare the performance of the NC gel presented in this thesis (without chemical crosslinker) to NC gels prepared with chemical crosslinkers.

4. Further experiments should be done to examine other rheological properties like elastic modulus, storage modulus, loss modulus, gel strength of NC gels. This will help broaden our knowledge about the characteristics and behaviour and limitations of the gel.

5. Further test should be carried out to examine the stability of these gels over longer periods of time at different temperatures, resistance to syneresis should be examined to determine application in reservoirs with higher temperature

6. The performance of NC gels in the presence of crude oil could be examined, different chalk core plugs with different wettability can be used to characterise the effect of wettability on the fracture plugging potential of NC gels

7. Nanocomposite gels could be tested in core plugs with different types of fracture pattern and size to help examine its range of application

8. The performance of nanocomposite gel as a disproportionate permeability reduction agent should be examined.

9. Experimental investigation on the performance of the NC gel system can be evaluated in other types of reservoirs like sandstones.

10. Experimental data characterising the gels and factors influencing gel performance could be acquired in order investigate the possibility of modelling the plugging performance of nanocomposite gels.

11. Further experiments could be carried out to provide more understanding of the causes of laponite flocculation in seawater. It can also be examined if seawater ionic composition could be modified to enable compatibility with NC gels in order to examine the possibility of a NC gel fracture treatment and a combined seawater EOR process for chalks.

Chapter 6   Conclusions

This thesis has examined NC gels based on various polymers and clays, their performance in deionized and seawater, the sensitivity of laponite to cations like calcium and potassium and chalk, the potential of various low molecular weight glycols as gelation retarders and finally the evaluation of nanocomposite gels as a fracture plugging agent and their performance compared to laponite gels. In this chapter, the main conclusions of this thesis as follows:

1. The following conclusions can be drawn from examining the interactions and characteristics between various types of polymer and clay prepared in deionized and seawater using tube tests.
    *   Laponite clay formed better NC gels than Bentonite clay in both deionized and seawater
    *   Bentonite clay did not readily disperse in deionized or seawater
    *   Bentonite clay did not interact with polymers to form gel
    *   Flocculated was observed when Laponite prepared in seawater at all concentrations
    *   Laponite-PEG gelant solutions interactions resulted in the production of shear induced gels
    *   Laponite-HPAM interactions generally form stronger gels than Laponite-Gellan gum interactions
    *   The strength of the Laponite based NC gels increased with increase in concentration

2. Based on the study of the sensitivity of laponite to cations like calcium and potassium in salts and chalk was studied and the following conclusion can be drawn from this study:
    *   Laponite reacted with calcium and potassium in salts to form more viscous gels
    *   Laponite reacted with chalk to form more viscous gels
    *   The viscosity of the gels formed by reaction of laponite and cations depends on the ionic concentration of the water

- Increasing the concentration of KCl, CaCl$_2$, and chalk in the laponite dispersion led to an increase in the strength of gels formed.

3. From the study of the sensitivity of laponite gels to gelation retarders was studied by adding different types of low molecular weight glycol. The following conclusions can be made from this study.
   - Butyl glycol did not retard the gelation process in laponite dispersions. An increase in concentration did not show any significant effect on the gelation time.
   - Butyl diglycol and PEG retarded the gelation process of laponite dispersions by slowing down laponite-laponite particle interaction and increased the gelation time.
   - Significant decrease in viscosity was observed for both Butyl diglycol and PEG when mixed with laponite in 1:1 weight ratio compared to when mixed in 2:1 weight ratio
   - PEG induced solutions provided longer gel times compared to Butyl diglycol induced solutions
   - The addition of PEG to laponite dispersions will not impair the optimum strength of the gel formed

4. The potential of nanocomposite gels as a fracture plugging agent in chalk was examined and compared to that of laponite gels. The following conclusions can be made from this study:
   - A reduction in the permeability of water from 106 md to 1.6 md and a corresponding residual resistance factor of 65.4 was recorded due to treatment of the fractured chalk with NC gel
   - The injection of laponite gels resulted in a permeability reduction from 22.1 md to 3.8 md and a corresponding residual resistance factor of 5.8
   - Nanocomposite gels showed good potential as a fracture plugging agent in chalks.

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