# TOWARDS WATER SUSTAINABILITY: UTILIZING OILFIELD PRODUCED WATER IN FORMULATING OIL AND GAS STIMULATION FLUIDS THROUGH INCORPORATION OF CHELATING AGENTS

BY

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*To the unsung hero of my life*

*To whom I owe all my success*

*To my mother*

*for her countless sacrifices*

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First and foremost, thank you god for your immense blessings, goodness and grace that is seen and felt in my everyday life. Secondly, I would like to express my sincerest gratitude for my family members and friends who provided me with tremendous support and hope throughout my time pursuing this degree. I would like to express my gratitude for my advisor Dr. Bassam Tawabini for his continuous guidance and unwavering support, my co-advisor Dr. Mohamed Mahmoud for his immense knowledge and help, my mentor Dr. Mohammed Bataweel for providing great mentorship and incredible support, and the committee members Dr. Korhan Ayranci and Dr. Svetoslav Georgiev for their valuable knowledge and feedback.

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| | | |
|---|---|---|
| **PW** | : | Produced Water |
| **DI Water** | : | Deionized water |
| **GLDA** | : | Glutamic Acid – Diacetic Acid |
| **CMHPG** | : | Carboxymethyl Hydroxypropyl Guar |
| **PPTG** | : | Pound per Thousand Gallons (Solids Concentration Unit) |
| **cP** | : | Centipoise (Viscosity Unit) |
| **TDS** | : | Total Dissolved Solids |
| **HPHT** | : | High Pressure High Temperature |
| **PPM** | : | Parts per Million |
| **API** | : | American Petroleum Institute |
| **RPM** | : | Revolution per Minute (Speed unit) |
| **CBM** | : | Coalbed methane |
| **GCC** | : | Gulf Cooperation Countries |
| **MENA** | : | Middle East and North Africa |

# ABSTRACT

Full Name : Mustafa Ahmed Alkhowaildi
Thesis Title : Towards Water Sustainability: Utilizing Oilfield Produced Water in Formulating Oil and Gas Stimulation Fluids through Incorporation of Chelating Agents
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Water scarcity is a highly pressing and accelerating issue facing our planet. There is a great incentive to develop sustainable solutions through reusing wastewater or produced water (PW), especially in nations where it is generated abundantly. In petroleum industry, PW represents the water produced as a by-product during the extraction of oil and gas operations. It is by far the largest wastewater stream within the industry with hundreds of millions of produced water barrels being generated per day worldwide. This research investigates a re-use opportunity for PW to replace the freshwater use in well stimulation applications within the oil industry. Introducing a certain environment-friendly chelating agents allowed formulating a fluid system that possesses the rheological properties of that in conventional fluid systems. A full rheological characterization is the main objective of this research, through the use of chelation chemistry, studying the effect of multiple parameters controlling the thickening profile of the fluid has enabled us to predict an optimum formulation to build a new stimulation fluid based on untreated oilfield produced water. The experiments were carried out using two viscometer equipment; Fann Model 35 and Chandler Model 5550, with the latter being the apparatus used for high pressure high temperature (HPHT) tests. Variables such as polymer concentration and pH are shown to have a great impact on the viscosity, while temperature and concentration of the chelating agents are shown to control the thickening profile as well as its stability and breakage behaviors. 50 pptg of polymer concertation (CMHPG- Carboxymethyl Hydroxypropyl Guar) in 20 wt. % chelation concentration (GLDA-

Glutamic acid, diacetic acid) is shown to sustain viscosity of 172 cP for nearly 2.5 hours at 150°F and 100 $S^{-1}$ shear rate. The newly developed fluid system, solely based on polymer, chelating agent, and PW has shown some rheological capabilities to replace the conventional stimulation fluids that are based on freshwater. The newly developed fluid can also have economical value realization due to containing much less additives compared to conventional fluids.

# ملخص الرسالة

**الاسم الكامل**: مصطفى أحمد مكي الخويلدي

**عنوان الرسالة**: نحو استدامة المياه: الاستفادة من المياه المنتجة من حقول النفط في تطوير موائع تحفيز النفط والغاز باستخدام مركبات مخلبيه

**التخصص:** علوم بيئية

**تاريخ الدرجة العلمية**: مايو ٢٠٢١ ميلادي، الموافق رمضان ١٤٤٢ هجري

ندرة المياه هي من القضايا الملحة والمتسارعة للغاية التي تواجه البشرية والمجتمع البيئي. هناك حافز كبير لتطوير حلول مستدامة من خلال إعادة استخدام المياه المنتجة من حقول النفط، خاصة في الدول التي تتولد فيها هذه المياه بكثرة. في أعمال البترول، تمثل هذه المياه أكبر منتج ثانوي أثناء عمليات استخراج النفط والغاز حيث يتم إنتاج مئات الملايين من براميل المياه يوميًا في جميع أنحاء العالم. هذه الرسالة تبحث عن فرصة إعادة الاستخدام هذه النوعية من المياه المنتجة لتحل محل المياه العذبة في أعمال تحفيز الإنتاج لأعيان الزيت والغاز الطبيعي. إدراج مركبات مخلبيه معينة وصديقة للبيئة أيضاً مكنتنا من تكوين مائع ذو خصائص انسيابية مشابهة للسوائل التقليدية حاليا. التوصيف الشامل لخصائص الانسيابية لهذا السائل هو الهدف الرئيسي من هذا البحث، فمن خلال استخدام المركبات المخلبيه ودراسة تأثير العوامل التي تتحكم في انسيابية السائل، أصبح من الممكن التنبؤ بالتركيبة المناسبة لهذا السائل المكون من المياه المنتجة. تم إجراء التجارب باستخدام جهازي قياس اللزوجة؛ فان ٣٥ وتشاندلر ٥٥٥٠، مع كون الثاني هو الجهاز المستخدم لاختبارات الضغط العالي ودرجات الحرارة المرتفعة. تظهر الاختبارات أن بعض العوامل مثل تركيز البوليمر والرقم الهيدروجيني للسائل لها تأثير كبير على اللزوجة، بينما تظهر درجة الحرارة وتركيز المركبات المخلبيه في التحكم في تغليظ السائل وكذلك في مدة ثبات وضعف هذه اللزوجة المكتسبة. أظهرت النتائج أن ٥٠ (رطل لكل ١٠٠٠ جالون) من البوليمر(CMHPG) في عينة ماء تحتوي ٢٠٪ من الوزن على مركب مخلبي (GLDA) حافظت على لزوجة بمقدار ١٧٢ سنتيبواز لمدة ساعتين ونصف تقريباً تحت درجة حرارة ١٥٠ درجة فهرنهايت وسرعة احتكاك ١٠٠ ١/ثانية. أظهر سائل الموائع المطور هذا، والذي يتكون فقط من البوليمر، ومركب مخلبي ، وعينة من المياه المنتجة، قدرات ريولوجية كبيرة تمكننا من استبدال سوائل التحفيز التقليدية التي تعتمد في تكوينها على المياه العذبة. يمكن أن يكون للسائل المطور حديثًا أيضًا قيمة اقتصادية نظرًا لاحتوائه على مواد مضافة أقل بكثير مقارنة بالسوائل التقليدية.

# CHAPTER 1

# INTRODUCTION

Water scarcity and depletion of freshwater resources is a global concern and among the most predominant environmental challenges of the 21st century. One of the most pivotal challenges is the enormous water consumption of humans. In addition, climate change is expected to bring an increase of 1.4°C - 5.8°C in the average global temperatures by 2100 according to the intergovernmental panel on climate change. Precipitation levels, therefore, are also expected to change, with varying magnitudes and directions across the globe [1]. Because the hydrological cycle is tightly connected with climate change, these changes will significantly affect the quality and availability of water. Concerns over the impact and consequences of climate change often dominates discussions on water challenges by both scientists and policy makers, however, the widespread water crisis is believed to be mostly linked to growing populations and the extensive consumption of water [2].

In light of the extreme stress on freshwater and underground water resources, one way of seeking alternatives is to utilize and recycle the enormous amount of generated produced water from the oil and gas industry to suffice some of the industry's own water-needing operations. In exploration and production (E&P), some of the processes associated with hydrocarbon recovery require huge quantity of freshwater, for example, the water usage in hydraulic fracturing operations in 2010 in the U.S. is estimated to be between 70 to 140 billion gallons of water. Reusing produced water in well stimulation operations has emerged as a win-win

proposition, transforming the industry's biggest waste product into a resource, with added benefits of reducing the environmental footprint [3]. However, this process involves some technical challenges that need to be addressed in order to formulate a fluid system that meet the performance requirements. Some of these challenges include meeting the required fluid viscosity at certain shear rates (i.e. 200 cP at shear rate of 100 1/s for a minimum of 2 hours) and having no precipitation or suspended solids that could damage the formation and exhibiting ability to carry fracturing sands (elasticity properties).

Harnessing energy from natural gas has emerged as key element in the energy mix, it is increasingly becoming an important source and a "bridge" to a low-carbon future while responding to the ever-rising global energy demand [4]. Natural gas is the least carbon intensive fossil fuel and environmentally cleaner than coal and oil. Natural gas is found in deep underground rock formations, most often than not, being trapped inside pores of extremely low permeable rocks, that often require formation stimulation/treatment in order for commercial natural gas to flow. Over the past decade, technologies such as horizontal drilling and hydraulic fracturing have enabled the excessive growth of the natural gas industry (i.e. shale gas) [5].

Well stimulation is a well intervention treatment performed on oil or gas well to enhance the productivity of the well and increase the flow of hydrocarbon into the wellbore. Generally, it is designed for formations exhibiting a very low permeability, known as tight formations.

Well stimulation techniques are categorized into two main types: hydraulic fracturing and matrix stimulation. Hydraulic fracturing process involves a pressure injection of frack fluid into the wellbore to the formation creating cracks within subsurface rock formation through which natural gas will be mobilized more freely. Matrix stimulation is a process that involves pumping acid nearby the wellbore region to dissolve minerals that could hinder well's productivity.

### 1.1 The Grand Challenge of Water:

Water scarcity is a highly pressing issue as highlighted by the World Economic Forum in its Global Risks 2019 report. It is thought to have one of the highest impacts and most likely risks facing the planet [6]. Water stress is defined as the ratio of total water withdrawals for different consumption purposes by a country relative to the available renewable surface water [7]. The severe water-scarcity threshold set by the United Nations is 500 cubic meters per capita per year [8]. The Gulf Cooperation Council (GCC) countries are all classified as water-scarce nations. According to the World Resources Institute [9], where they use a robust tools and peer reviewed methodology, 17 countries, mostly in the Middle East, face the risk of extremely high water-stress (Fig. 1). Due to the severe scarcity of water resources, Middle East and North Africa (MENA) regions will particularly be the most liable to climate effects on water resources [10].

### 1.2 The case for Saudi Arabia:

Saudi Arabia occupies most of the Arabian Peninsula. It is one of the largest arid countries without permanent rivers or lakes [11]. Located in the tropical and subtropical desert region of

the Middle East bonded by the Arabian Gulf and the Red Sea, temperatures can exceed 50°C in some areas, with overwhelmingly hot and dry conditions [11,12]. Having a limited underground water resources and almost non-existent surface water resources, most of its underground "fossil" water is ancient and dates back to between 13,000 and 17,000 years ago [13]. The fossil water in Saudi Arabia's aquifers is characterized as storage dominated rather than recharge-flux dominated, as only a very tiny fraction of ground water is recharged [14]. The groundwater has been under more pumping than what nature can replace through precipitation. It is estimated that the renewable internal freshwater resources per capita for Saudi Arabia is 72.9 cubic meters in 2017 compared to a world average of around 6000 cubic meters per capita, putting it amongst the poorest ten countries in terms of water resources per inhabitant [15].

Many years of irresponsible agricultural policies, rapid population growth and low tariff structure for water ($0.03/cubic meter) has led to an unsustainable culture of water usage in Saudi Arabia. Despite the very limited renewable water resources in the country, its water consumption levels is third only to the United States and Canada, with 250 liters of water used per capita daily [16,17].In recognition of its water-starved status, Saudi Arabia has taken many reformative actions towards its water consumption practices, providing a leading example for adaptation and mitigation, as recent reports demonstrated how the nation has converted more toward sustainable solutions [18,19].

Saudi Arabia serves as a global leader in development and use of desalination technologies, producing the largest volumes of desalinated water in the world and successfully replacing the

use of groundwater as the primary source of drinking water [20]. With 31 operating desalination plants, Saudi Arabia is the largest producer of desalinated water in the globe.

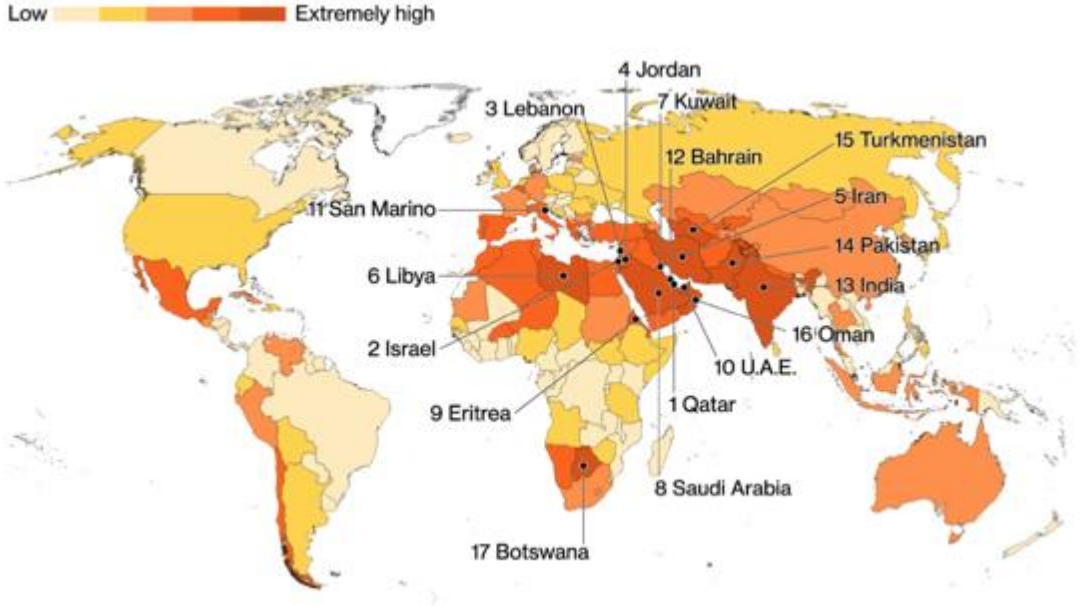


**Figure 1: Data on baseline water stress by each country (World Resources Institute).**

Well stimulation is a treatment technique used to generally enhance the productivity of oil and gas wells and increase the flow of hydrocarbon into the wellbore. It is done by using stimulation fluids that are generally water-based, comprises of 99% water and 1% chemical additives to meet the required properties of the fluid. Fracturing fluids must have the following characteristics: high proppant carrying capacity (viscosity), low pipe friction, low fluid loss (fluid efficiency), easy preparation and easy removal from the treated reservoir (clean up). The fluid can generally achieve very good viscosity by means of a gelling agent, guar-gum derivatives are usually used, such as hydroxypropyl guar (HPG) and carboxymethyl hydroxypropyl guar (CMHPG). Less residue and faster hydration is achieved using these gelling agents, however, they are sensitive to salts and solids content in the source water [21].

In this research, the chelating agent effect in thickening and breaking the stimulation fluid is an area that hasn't been widely investigated in the literature. The outcome of this research is a fluid formulation exhibiting an optimum rheology for stimulation applications in oil and gas. Replacing freshwater with high TDS PW will introduce technical challenges, the biggest one being achieving the required fluid rheology (i.e. 200 cP viscosity under 100 1/s shear rate, for 2 hours, under HPHT conditions) to fracture tight gas formations, as salts in water are known to hinder thickening behavior after adding polymeric gels. This research will introduce chelating agents to capture the salts present in PW and allow gelling agents to enhance the viscosity of the fluid. The concentration of chelating agents as well as polymeric gel that yielded the best rheology results and most optimum for stimulation purposes are shown in the results section.

## 1.3 Research Motivation

In recent years, amidst the rise for energy demand, natural gas from tight reservoirs has been targeted abundantly around the globe by different oil operators. Hydraulic fracturing technology has been instrumental for the successful exploitation of energy from tight formation, shale gas for example. However, the process is associated with enormous usage of freshwater resources. Hydraulic fracturing uses as little as 500,000 gallons of freshwater, but can reach up to 6 million gallons per well, depending on the type of the well and the number of stages stimulated.

There is a great incentive and growing interest for developing sustainable solutions through reusing wastewater or produced water (PW) for developing stimulation fluids, especially in countries where PW is generated abundantly and available for reuse. Developing a stimulation fluid based on PW will save millions of freshwater gallons per single well treatment, which an increase in its activity is seen recently.

## 1.4Research Objectives

The ultimate objective of this research is to conduct a rheology characterization to aid formulate an environment-friendly fluid system based on 100% PW and biodegradable material, capable of stimulating oil and gas formations to enhance its productivity. Additionally, propose a re-use opportunity for oilfield produced water that can contribute to conservation of freshwater resources. The following are the proposed research objectives:

1. Study the effect of chelating agents (GLDA-Glutamic acid, diacetic acid) on divalent cation $Ca^{++}$ present in oilfield produced water.
2. Determine the optimum concentrations for the polymeric gel (CMHPG-Carboxymethyl Hydroxypropyl Guar) and chelating agents (GLDA-Glutamic acid, diacetic acid) to formulate the most effective rheology using synthesized PW (TDS of 70,000 ppm & hardness 7,000 ppm) base fluid, under high pressure (300 psi) high temperature conditions (+150 F).
3. Study the effect of various variables such as temperature (150, 200, 300°F), shear rate and pH (4, 8 and 10) on the rheology of the fluid system.

# CHAPTER 2

# LITERATURE REVIEW

The literature review in this chapters covers mainly two parts: the oilfield produced water and well stimulation in petroleum industry. The PW part covers the overall characteristics of such complex stream and its common constituents. The well stimulation covers the industry's current practices and literature background for its development. Additionally, successful stories of utilizing such wastewater for stimulation applications are reported with the current research gap existed in the industry with regard to developing such fluid with chelating agents.

## 2.1 Oilfield Produced Water

Rocks in deep subsurface are often saturated with gas, oil or water, or sometimes combined together in a single reservoir. Most hydrocarbon-bearing rocks are believed to be once completely permeated with water before petroleum migration and trapping [22]. Primarily driven by buoyancy, the hydrocarbon fluids migrated to trap structures while pushing some of the water existed to turn the formation into a hydrocarbon reservoir. Thusly, petroleum reservoirs normally consist of water, but also may include fluxes from nearby zones, or originated from production activities such as injection or well stimulation. This water is normally referred to as "formation water" or "connate water" and categorized as produced water when the reservoir is produced and this water is pumped to the surface [23]. In oil and gas industry, produced water is any water stream that flows from an oil or gas producing reservoir. Produced water is considered by far the largest by-product waste associated with oil and gas exploration and production [24].

Produced water is brought to the surface with the hydrocarbons as a produced fluid mixture. The composition of this fluid mixture is directly related to the chemical characteristics of the formation it is produced from, and the type of hydrocarbon being produced i.e. crude oil or natural gas. However, produced water will generally include mixture of either liquid or gaseous hydrocarbon, dissolved or suspended solids, injected fluids or solids from production activities. Produced water properties and volumes can vary throughout the lifetime of a reservoir, and can dramatically change if water-flooding operations are conducted on the reservoir.

Water is generated from the extraction of oil and gas from nearly all types of wells, regardless if the hydrocarbon is extracted from onshore or offshore wells, conventional or unconventional sources including coalbed methane (CBM), tight sands and gas shales. However, natural gas wells typically produce lower volumes of water than oil wells, except in certain types of gas resources such as coalbed methane (CBM), mainly because of CBM produced mechanism that requires pumping large quantities of water.

Produced water accounts for approximately 98 per cent of the total generated waste volume by oilfield operations in the United States, it is by far the largest stream waste generated in the oil and gas industry. According to the American Petroleum Institute (API), around 18 billion barrels of produced water was generated in the year 1995 by US onshore operations alone. Additional large volumes of produced water is generated by US offshore operations, and from thousands of additional wells in other countries worldwide [23]. Khatib et al. [25] estimate that around 77 billion bbl of produced water was generated worldwide in the year of 1999. Dickhout et al. [26] estimated

that more than 70 billion bbl of produced water was generated in the year 2009, of which, 21 billion bbl is generated by the United States.

Produced water is not one component, rather varies from a simple product to a complex one. It is known to be a mixture of organic and inorganic chemicals. The chemical, physical and biological properties of produced water vary considerably depending on certain factors. These factors include the geographical location of the field, the geological formation, the reservoir age, type of produced hydrocarbon as well as the geochemical composition of the reservoir [23]. In addition, produced water properties and volume can vary throughout the lifetime of a reservoir.

The characteristics of produced water are also subjected to the type of chemicals used in extracting and processing hydrocarbons as well as the extracting method used. The compositions can differ in magnitude of concentration from one region to another, however, literature suggests that the major components of produced water is qualitatively constant from gas & oil wells across different fields [27]. The main components of produced water are categorized as follow [28]:

- Salts (expressed in total dissolved solids (TDS) or total conductivity)
- O&G (oil and grease)
- BTEX (benzene, toluene, ethylbenzene, and xylenes)
- PAHs (polyaromatic hydrocarbons)
- Phenols
- Organic acids
- Various natural organic and inorganic compounds (e.g. calcium, magnesium, sulphates)
- Chemical additives used in extraction process (e.g. corrosion inhibitors, biocides)

- NORM (naturally occurring radioactive material)

The concentrations of these components vary across different regions and reservoirs. Table 1 summarizes the range of these concentrations as gathered in literature [29]:

**Table 1. Generic PW Composition**

| **Parameter** | **Concentration (mg/l)** |
|---|---|
| **Major Parameters** | |
| TDS (Total dissolved solids) | 100–400,000 |
| TSS (Total suspended solids) | 1.2–1000 |
| COD (Chemical oxygen demand) | 1220–2600 |
| TOC (Total organic content) | 0–1500 |
| Total organic acids | 0.001–10000 |
| **Chemicals Additives** | |
| Glycol | 7.7–2000 |
| Corrosion inhibitor | 0.3–10 |
| Scale inhibitor | 0.2–30 |
| **BTEX** | |
| Benzene | 0.032–778.51 |
| Toluene | 0.058–5.86 |
| Ethylbenzene | 0.026–399.84 |
| Xylene | 0.01–1.29 |
| **Other pollutants** | |
| Saturated hydrocarbons | 17–30 |
| Total oil and grease | 2–560 |
| Phenol | 0.001–10,000 |
| **Metals** | |
| Na | 0–150,000 |
| Sr | 0–6250 |
| Zn | 0.01–35 |
| Li | 0.038–64 |
| Al | 0.4–410 |
| As | 0.002–11 |
| Ba | 0–850 |
| Cr | 0.002–1.1 |
| Fe | 0.1–1100 |
| Mn | 0.004–175 |
| K | 24–4300 |
| **Other ions** | |
| B | 5–95 |
| $Ca^{2+}$ | 0–74,000 |
| $SO4^{2-}$ | 0–15,000 |

| | |
|---|---|
| $Mg^{2+}$ | 8–6000 |
| $HCO3^{-}$ | 77–3,990 |
| $Cl^{-}$ | 0–270,000 |

**Salinity and total dissolved solids (TDS):**

The dissolved salts are considered the most predominant constituent in produced water. The salinity of produced water varies from 100 ppm to 300,000 ppm, which in most cases is much more saline than seawater that has an average salinity of about 35,000 ppm and that is the reason for produced water to generally be denser than seawater. The higher salinity values are resulting from the presence of more dissolved sodium and chloride, however, concentrations of calcium, potassium and magnesium are usually lower than that of seawater [30]. In a study done by Guerra et al. [31], TDS values in produced water compromised from higher concentrations of sodium and bicarbonates (Na, $HCO3^{-}$), than that of seawater.

TDS concentrations were found to be changing over time putting more challenges on the management and reuse of produced water. According to Rosenblum et al. [32], TDS concentrations vary for several reasons including the location of the well in their respective field as well as the differences of geological characteristics between basins. Furthermore, the concentration of TDS is found be different between conventional and unconventional wells. The concentration of TDS in coalbed methane wells is less than 50,000 mg/l while in conventional wells, it is reported to be as high as 400,000 mg/l [31].

**Inorganic ions, metals and NORM:**

Sodium and chloride ions are found to be the most abundant in produced water, whereas phosphate is found to have the lowest concentration between other salt ions. In both conventional and

unconventional wells, sodium is found to be the most predominant salt cation with 81 per cent in conventional wells, and 90 per cent in unconventional wells [31]. Moreover, produced water with high salinity, ions such as magnesium, chloride, sodium, sulphate, bromide, iodide, potassium, and bicarbonate are found abundantly. The sulphate and sulfide ions presence in produced water can lead to accumulation of insoluble sulphate and sulfide at high concentrations whereas the presence of bacteria can lead to a reduction in sulphate but in turn, presence of sulfides (e.g. hydrogen sulfide) [33].

Certain metals such as Fe, Ba, Cr, Zn, Ni, Pb, Cu and others may be found produced water, however, the type and concentration of these metals are influenced by the geological formation as well as the injected water volume and composition. The concentration of these metals can amount to 100 to $10^5$ times that found in seawater [28].

Naturally occurring radioactive material (NORM) are originated in the geological formations and are carried within produced water streams as dissolved solids and may precipitate into scale or sludge when temperature is reduced at surface. The most abundant NORM elements are $^{226}$Ra, $^{228}$Ra and Ba. The radioactivity of produced water is generally resulted from the radium that is co-precipitated with barium sulphate or other type of scales [34]. Up to 21 Bq/l of $^{228}$Ra has been detected in produced water in some oilfields [30].

**Total Suspended Solids (TSS), Chemical Oxygen Demand (COD) and Biological Oxygen Demand ($BOD_5$):**

The chemical oxygen demand (COD) is an indicative measure for the amount of oxygen that can be consumed by reactions in a solution, it is a very important measurement especially while

attempting to understand the environmental chemistry of the water body and its potential environmental impact in the case of discharging or reusing. Similarly, the biological oxygen demand (BOD) is the amount of dissolved oxygen required by aerobic biological organisms to break down the organic material present in a certain water sample. According to Fillo et al. and Johnson et al. [27,35], the estimated range of COD concentrations in produced water ranges from 2600 mg/l to 120,000 mg/l. Gomes et al. [36] found that COD is more precisely between 27,000 mg/l and 35,000 mg/l in samples collected from oilfields in the USA.

In natural gas fields, the BOD concentrations in produced water are reported in the range of 75-2870 mg/l [27]. Chemical additives and organic materials used in drilling fluids, and inorganic reduced elements such as iron and manganese can result in higher BOD values in produced water obtained from these particular wells. Dissolved oxygen in water bodies can severely deplete if received produced water with high concentrations of BOD and COD. Therefore, oxidization of these waste water streams is important to prevent contaminating natural waters.

Total suspended solids (TSS) include any drifting or floating materials found in a produced water stream, such as formation particles, silt, sand, sediment. In a study by Fillo and his colleagues [27], TSS concentration in produced water is between 14-800 mg/l and between 8 and 5484 mg/l in a study by USEPA [37]. Another study estimated the range of TSS in produced water to be 1.2-1000 mg/l [38].

**Dissolved Gasses:**

Oilfield produced waters contain large quantities of dissolved gasses, the most common gasses include volatile hydrocarbons, carbon dioxide, oxygen and hydrogen sulfide [39]. They are either formed naturally, by bacterial activities or by chemical reactions in water. The solubility of such gases in the water increases with pressure and decreases with salinity and temperature [28].

**Oil and Grease:**

Oil and grease concentrations in produced water is reported to be in the range of 6-60 mg/l, according to multiple studies [37,27,35]. PAHs and some of the heavier alkyl phenols such as $C_6$-$C_9$ alkyl phenols are found as small and suspended oil droplets (dispersed oil) because they are less-soluble in produced water [40].

**Organic Acids:**

Many organic acids are found in produced water such as dicarboxylic acids, monocarboxylic acids, in addition to other low molecular weight acids such as hexanoic acid, formic acid, acetic acid, butanoic acid, propanoic acid. Yet, the most abundant organic acids present in produced water are formic and acetic acids. The concentration of both formic and acetic acids were ranging from less than 70 mg/l to as high as 5735 mg/l from produced water from the Gulf of Mexico and Maria basin in the coast of California [41,42,43,44].

**BTEX and Phenols:**

BTEX and phenols are found in produced water as dissolved hydrocarbons while, as mentioned before, PAHs exist as dispersed oil in the solution. BTEX are volatile organics and low molecular weight aromatic compounds that are found in produced water, they are considered the most soluble

compounds in produced water[45]. Higher levels of benzene, toluene and ethylbenzene were detected in produced water samples from natural gas wells than in oil wells[27]. Benzene is considered to be the most predominant compound present in produced water, in a study carried out by [46], the concentration of benzene from water from Gulf of Mexico oil field was between 0.44-2.80 mg/l, followed by toluene, xylene and ethylbenzene. The findings were similar in another study by Neff et al. [33]. However, in the same study done by Dorea et al. [46], he found benzene concentrations to be in the range of 1.5-779 mg/l in produced water collected form the Permian basin. Similarly, it was also the highest but this time followed by ethylbenzene, xylene and toluene.

Phenols are class of aromatic organic compounds consisting of a hydroxyl group attached to an aromatic hydrocarbon group. Concentrations of phenols in produced water vary from gas to oil producing wells, however, produced water from gas condensate wells are thought to be with the highest concentrations of phenols [29]. Phenols concentrations in produced water could reach up to 10,000 mg/l [34].

**Added Chemicals:**

Aside from its natural components, produced water may contain a numerous added chemical from the process of drilling or stimulation of the well, or from process of treating and separation of oil-gas-water, or from prevention of operational problems such as corrosion and hydrate formation. These substances are unique for every process and vary along the various production systems. Some of these chemicals are scale, corrosion and hydration inhibitors, biocides, asphaltene dispersants, defoamers, emulsion breakers, coagulants, flocculants …etc [29].

## 2.2 Well Stimulation

Well stimulation is a well intervention treatment performed on oil or gas well to enhance the productivity of the well and increase the flow of hydrocarbon into the wellbore. Generally, it is designed for formations exhibiting a very low permeability, known as tight formations. Well stimulation techniques are categorized into two main types: hydraulic fracturing and matrix stimulation. Hydraulic fracturing process involves a pressure injection of frack fluid into the wellbore to the formation creating cracks within subsurface rock formation through which natural gas will be mobilized more freely. Matrix stimulation is also associated with pumping fluid into the wellbore but without exceeding the rock fracturing pressure, designed to treat the near-wellbore reservoir formation by dissolving materials plugging the pores using acid or solvents.

Fracturing fluids are generally water-based, comprises of 99% water and 1% chemical additives to meet the required properties of the fluid. Fracturing fluids must have the following characteristics: high proppant carrying capacity (viscosity), low pipe friction, low fluid loss (fluid efficiency), easy preparation and easy removal from the treated reservoir (clean up). The fluid can generally achieve very good viscosity by means of a gelling agent, guar-gum derivatives are usually used, such as hydroxypropyl guar (HPG) and carboxymethyl-hydroxypropyl guar (CMHPG). Less residue and faster hydration is achieved using these gelling agents, however, they are sensitive to salts and solids content in the source water [21]. Cross-linkers such as borate (B), zirconium (Zr) and titanium (Ti), are used to generate a significant viscosity increase in the guar-polymer gels. The cross-linked fluids exhibit far better capacity to carry proppant than uncross-linked fluids. The viscosity measured by a rheometer in which fluid is tested under specific temperature and shear rate conditions is translated to carrying capacity of fluid. A minimum

transport capacity of 400 cP at 40 1/sec is preferred [47]. Since produced water composition is different than freshwater, the rheological effects on parameters pertinent to its composition will be evaluated from reported and published literature. These parameters include: pH, ionic strength, hydration time, and sulphate presence.

**pH:**

Produced water is known to be acidic with general pH values ranging from 5.1-6.8. Huang et al. [48] performed viscosity test for a 30-lb CMHPG gel, zirconium-based crosslinker, with 40 minutes hydration at different pH values followed by crosslinking. A 5.7 pH value performed the best achieving a maximum apparent viscosity of 2500 cp. These results were consistent with the findings of Rose et al. [49]. In the same study of Rose et al. [49], they were able to demonstrate that in polymer micro gels, Zr-dimmers dominates at pH 6 while cyclic-tetramers were found to be abundant at 7 pH. In the case of tetramers dominance and other large aggregates, the zirconium-based crosslinker function can be reduced leading to lower viscosity [50]. Furthermore, it was reported in multiple studies that the pH values were adjusted using a buffer solutions prior to having desired rheological parameters [51,52]. Through the study of molecular modeling, some studies on the CMHPG chains were found to be de-protonated at acidic settings (e.g. pH 4.6), indicating the adverse effect on the process of crosslinking [53].

**The hydration time:**

It is known that produced water contains high ionic concentrations, and that make it a poor solvent for guar-gum gelling agents such as CMHPG. Proper hydration time is therefore critical to achieve the desired rheological characteristics. In a study by Huang et al. [48], attempting to test the

feasibility of produced water as a base fluid for fracturing, the hydration time variable was tested and its effect on apparent viscosity were reported. It was observed that maximum apparent viscosity was achieved with longer hydration periods. The hydrations were performed prior to crosslinking the 30-lb CMHPG gel. There was a clear strong correlation between the maximum achieved apparent viscosity and hydration period between 60 to 120 minutes. This is to confirm that dispersion of polymer aggregates is reflective to having better viscosities over longer periods of hydration. Furthermore, the aggregation of CMHPG in the initial hydration stages was regularly witnessed during gel preparation. Since dispersion mechanism is gradual, increasing hydration time will therefore allow more solvated CMHPG concentrations in produced water and ultimately achieve higher gel viscosities after crosslinking.

In a study by Kakadijan et al. [54], where they used a zirconate-crosslinked 25 lb/1000 gals CMHPG gels formulated in untreated produced water. Hydration time of 3 and 9 minutes were performed, after 3 minutes of hydration the viscosity observed to have increased as the hydration temperature increased from 40° to 70°F. Whereas, after 9 minutes of hydration, the maximum viscosity was measured at 50°F, identifying effects of temperature and hydration time on viscosity. In addition, when the rheological profile of CMHPG cross-linked gel system (25 lb/1000 gals) was plotted against various temperatures from 240°F down to 180°F (simulating the cool down conditions of the Bakken formation). Contrary to the borate cross-linked Guar gel systems in freshwater, where a sharp decline in the rheological profile is observed at low temperatures when formulations are optimized for higher temperature conditions, this produced water formulated-fluid showed higher viscosity values as temperature descended. In addition, the presence of boron in produced water may introduce an early crosslinking and interfere with the correct hydration of

the polymer. It was observed that the addition of a chelating agent prevented the premature crosslinking and enabled further hydration of the polymer [52].

**Ionic strength:**

Produced water contains a various species of salts, the amount of ionic composition differs from one source of produced water to another. Examining the rheological performance on cross-linked gels then becomes important. There was no clear impact of ionic strength on the achieved viscosity of cross-linked 30-lb CMHPG gel with pH 5.5 and hydration time of 40 min [48]. However, it was observed that having different salt compositions but an identical ionic strength will yield similar viscosity values. Some polymers that are used in water based fracturing fluids like acrylamide or methacrylamide polymers, are known to be sensitive to high ionic strength, loss of viscosifying power and precipitation can occur [55].Cheremisinoff et al. [56] reported in a study supporting the use of saltwater for hydraulic fracturing. It is stated that even though the industry for a long time has operated on the idea that TDS is the bottleneck in common water reuse for fracturing, it is shown that TSS is the real challenge. Despite the very small size colloidal particles of TSS, they exhibit a huge surface area, making them abrasive friction inducers, negatively influencing the gelling hydration. And hence, low concentrations of TSS, not TDS, is what made freshwater favorable for gelling hydration used in hydraulic fracturing [56].

**Presence of sulphate:**

One major difference between freshwater and produced water is the high concentration of sulfate associated with produced water.

High sulfate concentrations in produced water may hinder persulfate self-decomposition causing more free radicals of sulfate for polymer breaking and therefore, a pre-mature breaking of the gel may occur. The magnitude of self-decomposition of persulfate was calculated mimicking chemical reactions of gelation conditions and time of a typical stimulation time (2 hours) with ionic strength similar to that of produced water. The results showed only a 0.7 per cent reduction in the persulfate concentration by self-decomposition within two hours, therefore, suggesting that sulfate concentration have minimal influence on the gel breaking process by persulfate [57]. Similar results were obtained in Huang et al. study [48], where no enhancement of gel breaking is seen with increased amounts of sulfates in produced water. In addition, CMHPG in several studies has shown better clean up behaviors downhole with minimum residue left and formation damage.

**Reported success stories in utilizing PW for stimulation:**

Marcellus formation produced water (TDS approximately 270,000 ppm) was used as the base-fluid with 50 gpt polymer, 6 gpt surfactant and 5 ppt encapsulated breaker without any crosslinkers or other additives. It has been observed that it can be mixed in high-salinity produced water as fracturing fluid without compromising its performance. The fluid has shown to have adequate rheology with both Marcellus and Bakken produced water in the US. The initial regain conductivity was about 71% and after gas cleanup, the final regain conductivity was 99%. [55]

In a study by Li et al. [58], it was found that Zr cross-linked CMHPG-guar polymer fluids have been successfully formulated with produced water and executed in field operations with reservoir temperatures of more than 250° F, with produced water TDS in the range of 280,000 mg/L and hardness levels of 91,000 mg/L. The study also reported that Zr and Ti cross-linked guar fluids mixed in produced water successfully attempted in wells with temperatures of 250°F, produced

water TDS around 300,000 mg/L and hardness levels of 44,000 mg/L. It is thought seawater behaves similarly to produced water as far as preparing cross-linked hydraulic fracturing fluids is concerned with the exception of having slightly higher concentrations of magnesium ions in seawater. Hence, seawater can be viewed as a sub-category of produced water. The produced water used in these studies were mostly untreated or had a very minimal treatment without changing the monovalent and divalent ion concentrations of the water.

## 2.3 Research Gap

There are many exerted efforts in utilizing and re-using wastewater in the oil and gas industry, however every single use has its distinctive criteria that dictate the performance requirement. In hydraulic fracturing operations, the freshness of the water is very critical in order to achieve the required fluid viscosity through introducing gelling agents (i.e. polymers), however this fluid thickening behavior is hindered in high TDS water systems. The following are some of the efforts that have been studied in the industry:

- Many studies have chosen to adjust to high TDS water systems by resorting to special types of polymers (usually synthesized and expensive) that are resistant to salts.
- Some studies have kept the same conventional fracturing fluid systems but introduced small amount of chelating agents to limit the salt effects.
- Some Studies investigated replacing all conventional fracturing fluids' additives by introducing special type of chelating agent that is thought of as multi-purpose agent (eliminating conventional additives such as cross-linker, breaker, biocide and stabilizer), however these studies have focused on seawater [59] and not oilfield PW which typically has higher hardness values.

The characteristics of oilfield PW differs from seawater mainly in the hardness (divalent ions i.e. calcium and magnesium), in this research we will investigate the possibility of formulating a new stimulation fluid based on 100% oilfield PW and without the use of numerous additives usually present in conventional fracturing fluid systems by introducing chelating agents.

Future studies can focus on other aspects of this fluid by conducting core-flooding experiments which can potentially add significant amount of knowledge in the overall effectiveness of this fluid as well as the post clean up behavior.

# CHAPTER 3

# METHODOLOGY

In this chapter, the experimental methodology will be discussed in details with the used materials, chemicals and instruments. Chemical structures and instruments capabilities, limitations and specifications are stated with each experiment objective explained.

The main objective in this research is to characterize the rheological properties of the developed stimulation fluid under different parameter variations. It is crucial to understand the effect of the various parameters controlling the rheological profiles of the fluid and its effectiveness in carrying out a specific application functions, in this research: stimulation of oil and gas wells.

The fluid is formulated using different concentrations of chelating agents: GLDA-Glutamic acid, diacetic acid, and polymer: CMHPG- Carboxymethyl Hydroxypropyl Guar, diluted in synthesized produced water. The fluid is then subjected to high pressure, high temperature conditions and various shear rates to study the stability and rheological properties of the developed fluid.

The pH system, additives concentration, shear rates, and temperature were the studied variables. For the pH, three different systems were tested to investigate the fluid's versatility under different pH systems: acidic, neutral and alkaline (pH = 4, 8 and 12). The conditions for shear rate were chosen at high and low shear rate values to capture the fluid's movement inside the wellbore of the well (low shear rate) and then inside the formation and fractures (high shear rate), the values

of shear rate are: 100, 170 and 511 $s^{-1}$. The additives concentration values were chosen based on industry's current practices and reported studies in the literature covering detailed studies on each additive. The temperature values are ranging from room temperature to high gas well temperatures in common fields (300°F).

The following steps were followed in carrying out this research to provide insight on each objective previously stated:

1. Test the effect of chelates on produced water:
   - Linear viscosity measurements for freshwater with gelling agent
   - Linear viscosity measurements for synthesized PW with gelling agent.
   - Repeat the experiments but with addition of salt concentration ($Ca^{++}$, $Na^{+}$, $Cl^{-}$, $Mg^{++}$) and observe the effect.

**Table 2: Chelating agents' effect on different water salt composition (viscosity)**

| # | Polymer Type/Concentration | GLDA type/Concentration (vol %) | Water salt composition (ppm) | Temperature (°F) | Shear Rate ($s^{-1}$) | pH |
|---|---|---|---|---|---|---|
| 1 | CMHPG (40 pptg) | GLDA (4%) | Freshwater | 68 | 170 | 4 |
| 2 | CMHPG (40 pptg) | GLDA (4%) | $Ca^{++}$ 4000 ppm | 68 | 170 | 4 |
| 3 | CMHPG (40 pptg) | GLDA (4%) | $Ca^{++}$ 6000 ppm | 68 | 170 | 4 |
| 4 | CMHPG (40 pptg) | GLDA (4%) | $Ca^{++}$ 8000 ppm | 68 | 170 | 4 |
| 5 | CMHPG (40 pptg) | GLDA (4%) | $Ca^{++}$ 10,000 ppm | 68 | 170 | 4 |
| 6 | CMHPG (40 pptg) | GLDA (4%) | TDS 60,000 ppm | 68 | 170 | 4 |

2. Find the optimum polymer concentration:

- Test the viscosity measurements of the commonly used concentrations industrial applications: 30 lb/1000 gals, 40 lb/1000 gals and 50 lb/1000 gals.

**Table 3: Polymer concentration optimization**

| # | Polymer Concentration | GLDA type/Concentration (vol %) | Temperature (°F) | Pressure (psia) | Shear Rate ($s^{-1}$) | pH |
|---|---|---|---|---|---|---|
| 1 | 30 pptg | GLDA (10%) | 150 | 500 | 100 | 8 |
| 2 | 40 pptg | GLDA (10%) | 150 | 500 | 100 | 8 |
| 3 | 50 pptg | GLDA (10%) | 150 | 500 | 100 | 8 |

3. Find the optimum chelating agent concentrations:

   - Test the viscosity measurements of the following chelating agent concentrations: 4%, 10%, 20%.

**Table 4: Chelating agent (GLDA) concentration optimization**

| # | Polymer Concentration | GLDA type/Concentration (vol %) | Temperature (°F) | Pressure (psia) | Shear Rate ($s^{-1}$) | pH |
|---|---|---|---|---|---|---|
| 1 | 50 pptg | GLDA (4%) | 150 | 500 | 100 | 8 |
| 2 | 50 pptg | GLDA (10%) | 150 | 500 | 100 | 8 |
| 3 | 50 pptg | GLDA (20%) | 150 | 500 | 100 | 8 |

4. Study the effect of the following parameters on the viscosity profile of the optimum developed fluid:

   - Temperature
   - pH
   - Shear rate

**Table 5: Variable testing conditions**

| Variable | pH | Temperature (°F) | Shear Rate ($s^{-1}$) |
|---|---|---|---|
| 1 | 4 | 150 | 100 |
| 2 | 8 | 200 | 170 |
| 3 | 12 | 300 | 511 |

### 3.1 Materials & Equipment:

The following are the materials, chemicals and equipment used to run the experiments:

- DI Water.
- Oilfield Produced Water Salts:
    - ◊ Sodium Chloride (NaCl)
    - ◊ Calcium Chloride ($CaCl_2.2H_2O$)
    - ◊ Magnesium Chloride ($MgCl_2.6H_2O$)
- Low pH GLDA (Glutamic Acid – Diacetic Acid Chelating Agent) pH: 3-4 with active agent concentration of 40 wt %. Manufactured by Nouryon, based in the Netherlands.
- High pH GLDA (Glutamic Acid, N, N-diacetic acid, tetrasodium salt) pH: 11-12 with active agent concentration of 47 wt %. Manufactured by Nouryon, based in the Netherlands.
- CMHPG Polymer (Carboxymethyl Hydroxypropyl Guar). Supplied by service provider companies in the kingdom.
- NaOH (solid pellets).

**Laboratory Synthesized Produced Water:**

In the literature, it is a widely accepted notion that PW composition vary considerably from a geographic place to another, from one field to another, and in some cases from one well to another. In order to fixate the composition for our testing, a collection of PW geochemical analysis is analyzed. The following is a generic representation of PW composition, consisting of 70,000 ppm total dissolved ions, the following salt recipe is used to synthesize the water to be used across all laboratory work as listed in Table 6 below:

**Table 6: Salt composition in synthesized PW**

| Salts | g/L |
|---|---|
| NaCl, g/L | 48.6 |
| $CaCl_2.2H_2O$, g/L | 22 |
| $MgCl_2.6H_2O$, g/L | 8.4 |

## Chelating agents:

According to the literature, several chelating agents were tested for formation damage after core-flooding experiments and the only chelating agent to have left minimal damage in the formation, required no breaker, and showed excellent stability properties, is the GLDA, it is also considered a readily biodegradable, therefore environment-friendly. The **GLDA** (**Glutamic Acid - Diacetic Acid Chelating Agent**) was used as chelating agent in this research (Fig, 2).


**Figure 2: High pH GLDA (left) and low pH GLDA (right)**

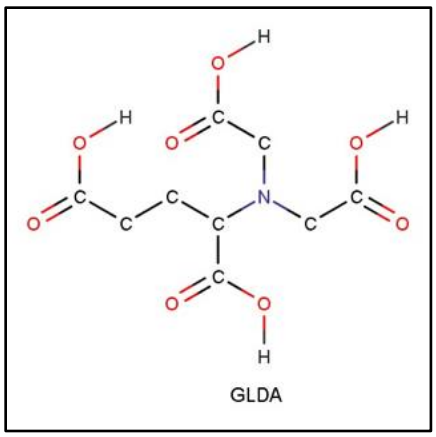


**Figure 3: Chemical structure of low pH GLDA**

## Gelling Agent:

The following polymer CMHPG - Carboxymethyl Hydroxypropyl Guar, **Fig. 4 & 5**, is a bio polymer, a guar gum derivative that can sustain high temperature reservoirs +300°F, and it is widely used in the industry. The selection of this polymer type was on basis of practicality and ease of deployment in industrial applications.

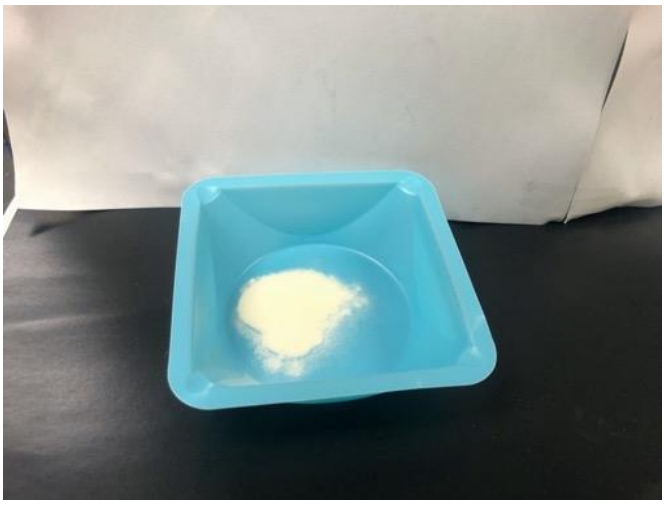

**Figure 4: CMHPG Polymer pre-hydration**

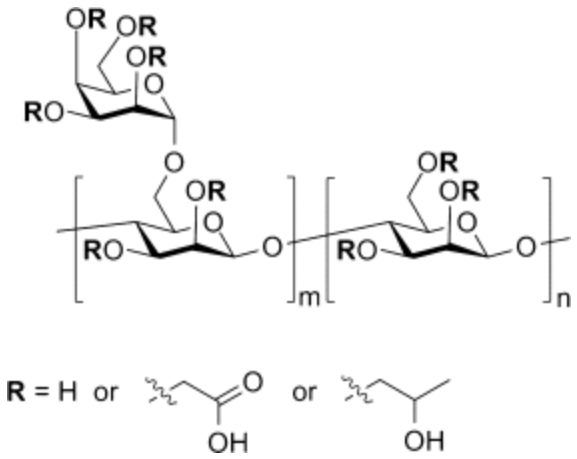


**Figure 5: Chemical structure of CMHPG**

The following are the equipment used to measure rheological profile of the developed fluid. Mainly two viscometer apparatuses were used in carrying out the laboratory work: Fann Model 35 (**Fig. 6**), mainly used to measure fluid viscosity at ambient conditions, and Chandler Model 5550 HPHT Viscometer (**Fig. 7**), used to assess the rheological profile of the developed fluid under elevated conditions.

**Figure 6: Fann Model 35 Viscometer**


**Figure 7: Chandler Model 5550 Viscometer**

- **Fann Model 35:**

Widely known and used in the oil industry for ambient viscosity measurements. Manufactured in Texas, USA, this instrument uses a rotating outer cylinder and bob, a simple design yet with accurate readings. It offers six different speeds: 600, 300, 200, 100, 6, 3 rpm.

- **Chandler Model 5550:**

This rotational viscometer offers excellent testing capabilities under HPHT, manufactured in Oklahoma, USA, it is a concentric cylinder viscometer that uses rotor and bob geometry designed for API specifications and requirements, and accepted by the energy industry. It offers a maximum temperature of 500°F and maximum pressure of 2000 psi and range of shear rate from 0.17 to 1700 s-1 with accuracy of ± 0.1 rpm. The equipment was calibrated prior to running experiments with equipment special fluids and standard deviation for the shear stress was recorded at 3 dyne/cm.

The rheological tests are intended to measure the apparent viscosity of the fluid system while being subjected to high temperature, high pressure and certain shear rates. The direct effect of polymer concentration, chelating agent concentration, and pH are thoroughly studied and analyzed. The following are the experiment design parameters:

1. A total of 100 mL is prepared using the base fluid is prepared in the lab using deionized water and salt concentrations as shown in **Table 6**.
2. The addition chelating agents takes place first and then mixed in a stirrer with synthesized PW, **Fig. 9 & 10**.
3. NaOH is used control the pH of the chelating agent used; a 2.3 g is added to 25 mL to increase the pH from 4 to 8, using a weighing balance, **Fig. 8**.
4. The addition of the polymeric powder then takes place, allowed to be hydrated and mixed for 10 minutes at least.
5. 52 mL of the mixed sample will be taken and placed in the Chandler rheometer cup, **Fig. 11**.
6. The pressure and temperature will be raised to the point of interest.
7. The shear rate and the running time will be determined on the software of the apparatus prior to starting the test.
8. Record the findings after the run is over and let the equipment cool down for properly cleaning up the cup from the residue of the sample before running any experiment again following the same steps mentioned.

**Figure 8: Digital weighing balance.**


**Figure 9: Magnetic stirrer**

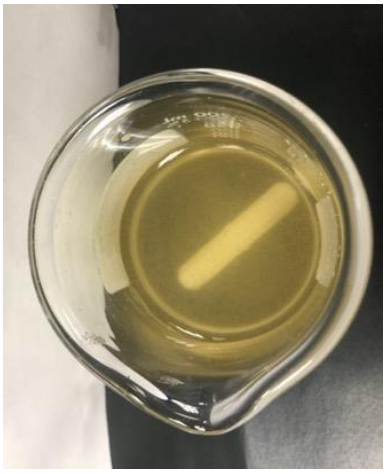

**Figure 10: While mixing fluid of 10 wt. % GLDA, 50 pptg CMHPG, PW**


**Figure 11: Prepared mixed fluid - 10 wt. % GLDA, 50 pptg CMHPG, PW.**

# CHAPTER 4

# RESULTS AND DISCUSSION

In this chapter the results will be discussed and thoroughly analyzed and compared to the existing literature that has discussed similar concepts. The initial results acquired from testing the fluid under ambient conditions showed clear chelation effect for major divalent cations. The HPHT tests provided insight on the breakage and stability of the fluid. The neutral pH system and highest concentrations of both additives (CMHPG and GLDA) showed the highest viscosity values. The fluid developed was also shown to be broken after some time (depending on the variables) which came in alignment with several previously conducted studies on incorporating GDLA additives in stimulation fluids, fracturing applications in specific.

## 4.1 Ambient Conditions and Baseline Experiments:

Initial baseline experiments were performed to establish an understanding of how the polymer hydrates in different water systems. It is widely mentioned in the literature that guar gum polymers hydrate better in freshwater systems and that dissolved ions tends to hinder the thickening behavior. This has been reaffirmed with clear observation in the lab using our 70k TDS PW and CMHPG polymer alone. In **Fig. 12**, deionized water is mixed with 30 lb/1000 gals CMHPG, showing an apparent viscosity of 54 cP under 170.3 $s^{-1}$, while the PW's apparent viscosity was measured at 42 cP under the same shear rate and polymer concentration, nearly a 22% reduction in apparent viscosity due to the presence of TDS in the water. The experiment was conducted across multiple polymer concentrations and the reduction in apparent viscosity remained visible, however with varying percentages. The hydration of the polymer is therefore a function of how

much ions are present in the water phase, it is well understood that water freshness is seen to be controlling the effectiveness of gelation buildup in this polymer type.

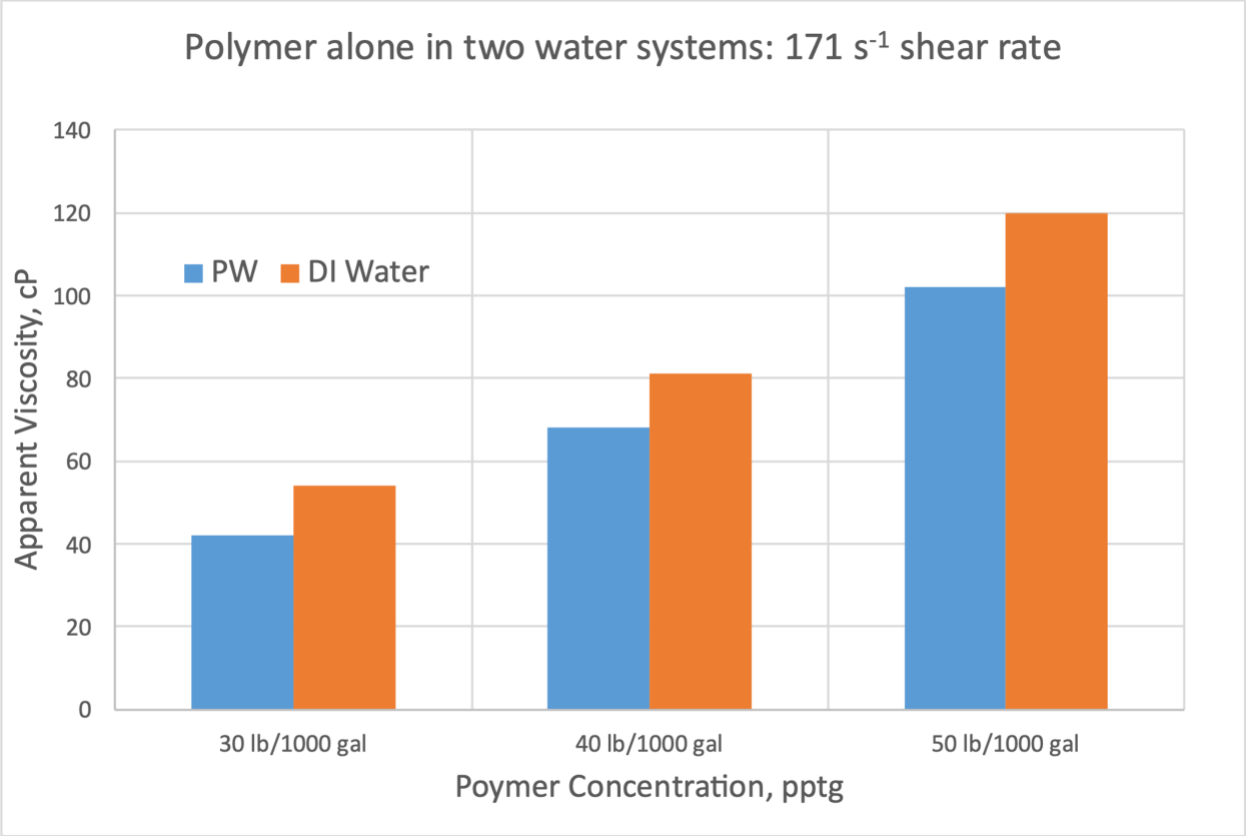


**Figure 12: Viscosity measurements of only polymer in DI and PW water**

In another baseline experiment, similar to what has been done on determining the water-softening effect of 4 wt. %, this time 10 wt. % of GLDA was introduced to a 70k TDS PW and hydrated with the same amount of CMHPG polymer 40 lb/1000 gals. The test measuring the apparent viscosity was done on the Fann Model 35, under ambient conditions and shear rate of 170.3 $s^{-1}$. The results showed an increase in the apparent viscosity in solution containing 10 wt. % GLDA, indicating strong effect in minimizing the TDS effect in forming a thickened gel. As shown in **Fig. 13**, with 10 wt. % GLDA, the viscosity was 93 cP, 27% more than solutions containing no GLDA,

with 68 cP. These results align with the literature in specifying GLDA as having an affinity to chelate Ca and Mg ions in the water, therefore allowing more free water to hydrate the polymer.

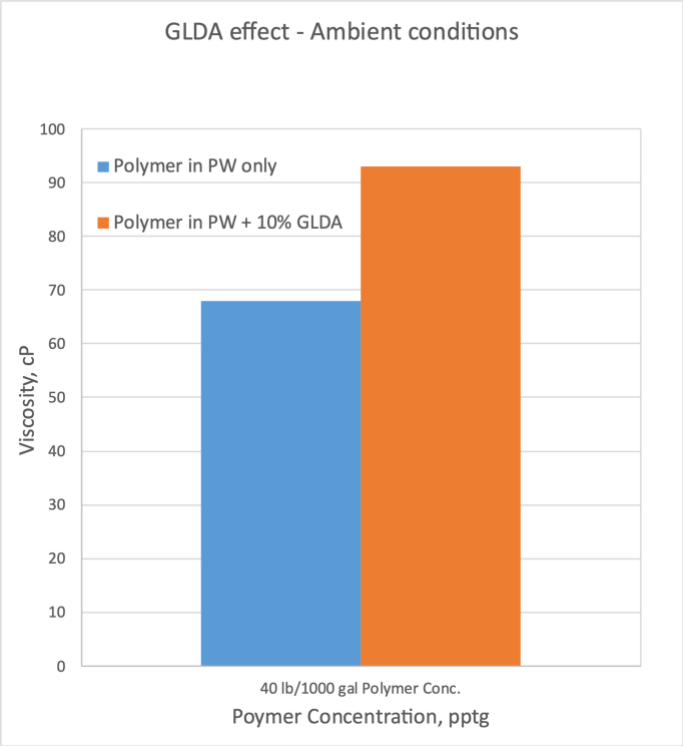


**Figure 13: The effect of GLDA addition to viscosity measurement**

## 4.2 Water-softening effects of 4 wt. % GLDA:

Initial experimenting was done to study the effectiveness of water-softening abilities of the GLDA on oilfield produced water and its most common divalent ions. The effectiveness of is simply determined through the ability of water to hydrate the polymer and build a viscous gel, where it is normally hindered in high TDS water systems, or produced water exhibiting high hardness profiles (presence of Calcium and Magnesium ions).

The 4 wt. % of GLDA has shown to chelate or capture most divalent ions present in the water systems, with up to 10,000 ppm of Ca++ dissolved in solution. This was observed when comparing

and showing similar values of viscosity of water containing various concentrations of $Ca^{++}$ compared to that in deionized water (**Fig. 14**).

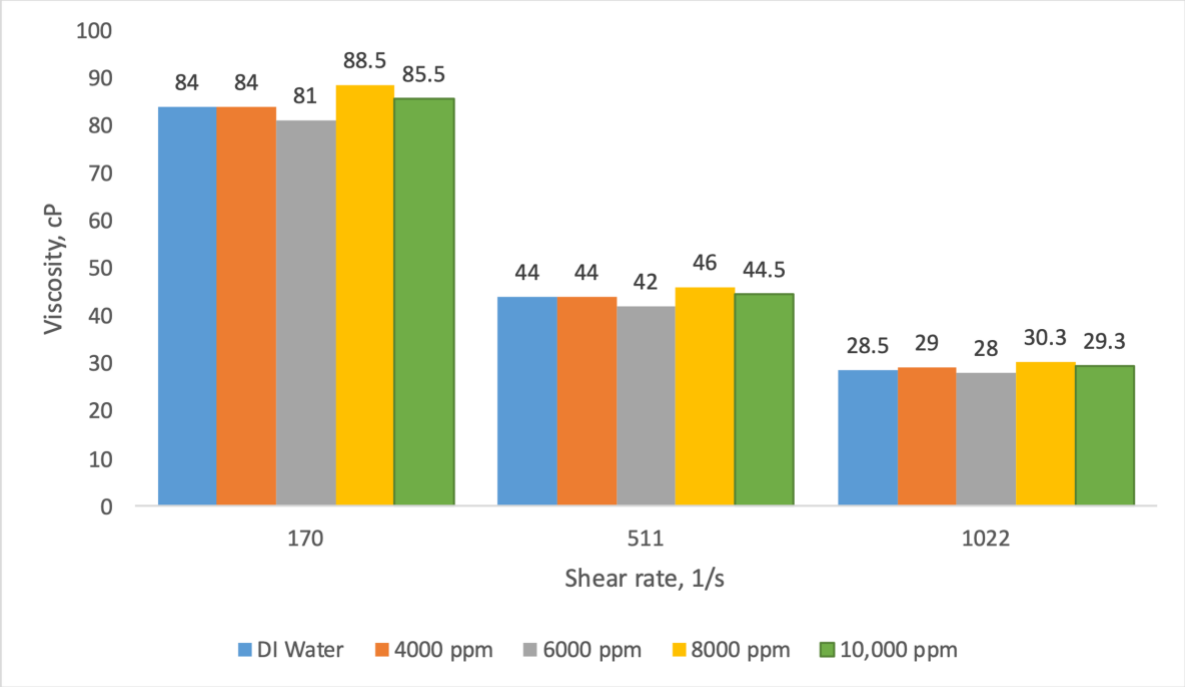


**Figure 14: Viscosity measurements under 3 different shear rates for 5 water systems**

Various concentrations of calcium ions ($Ca^{++}$) were dissolved in the water: 4000, 6000, 8000 and 10,000 ppm with 45 lb/1000 gals CMHPG polymer concentration. A minimum of 10 minutes' hydration was allowed on all samples before using the Fann Model 35 to measure viscosities across different shear rates at ambient conditions. As shown in the graph, similar values of viscosity indicate the successful chelation effect of 4 wt. % of GLDA to water containing up to 10,000 ppm of divalent ions, preventing the interruption of thickening behavior normally seen without the presence of chelating agents.

## 4.3 pH Variation:

50 lb/1000 gals of CMHPG polymer was mixed in PW solution, with 10 wt. % GLDA, the apparent viscosity was measured against time. The temperature was fixed at 150°F, the pressure at 500 psi

and the shear rate at 100 $s^{-1}$. Holding these parameters constant and running the experiment across three different pH systems (acidic = 4, neutral = 7.5 and basic = 12) showed that the neutral solution resulted in the best apparent viscosity read, slightly outperforming the acidic solution. As shown in **Fig. 15**, the neutral pH system maintained a constant viscosity at around 112 cP, the acidic pH system maintained a slightly decreasing viscosity at around 106 cP, while the basic pH system was reading at 82 cP. It is clear that the pH has an effect on the thickening behavior of the mixture and its stability with pH of 7-8 showing the most favorable conditions. It is worthy to mention that the basic pH systems take a longer time (5-6 hours) to hydrate the polymer and build up a viscous fluid, as opposed to almost instantly in other systems.

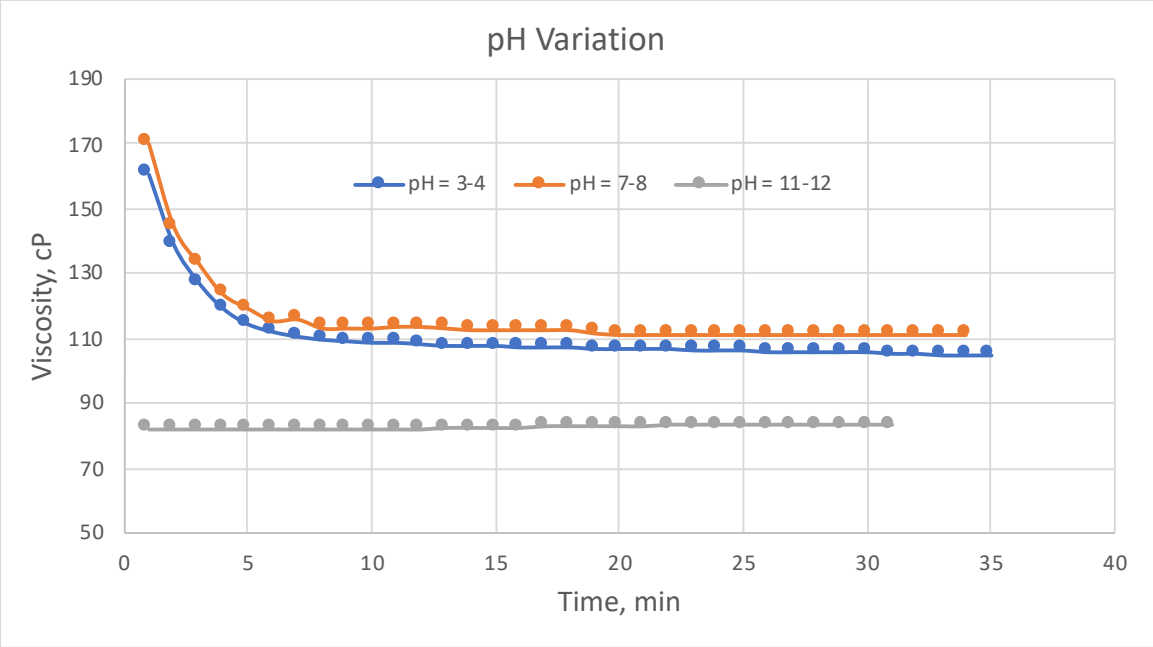


**Figure 15: Viscosity measurement over time for 3 different pH systems under 100 shear rate, 150 F and 10 wt. % GLDA and 50 pptg CMHPG conc.**

## 4.4 GLDA Concentration Variation:

50 lb/1000 gals of CMHPG polymer was mixed in PW solution, in an adjusted pH of 7.5 for the chelating agents, the apparent viscosity was measured against time. The temperature was fixed at 150°F, the pressure at 500 psi and the shear rate at 100 $s^{-1}$. Holding these parameters constant and running the experiment across two different GLDA concentrations systems (10 wt. % and 20 wt. %), the results, in **Fig. 16**, showed that the higher concentration of active chelating agents present in the system the higher apparent viscosity readings, which indicates that the more GLDA made available in the system the more thickening occurs with the polymer, assuming that only a certain amount of GLDA is held for capturing the ions in the system.

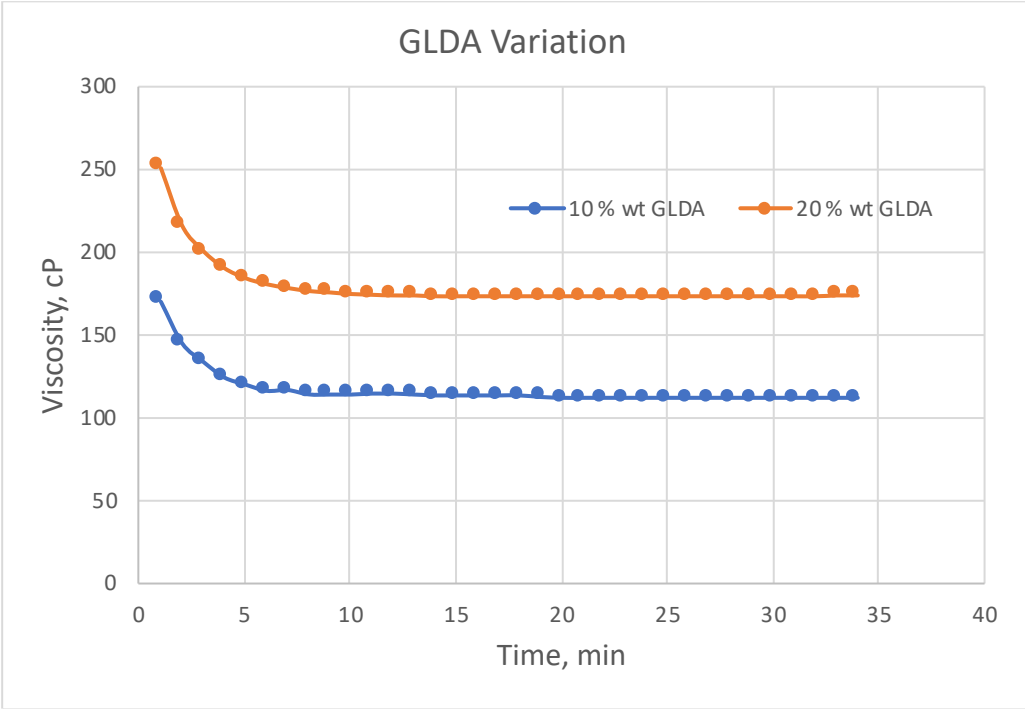


**Figure 16: Viscosity measurement over time for multiple GLDA concentrations under 100 shear rate, 150 F and 8 pH and 50 pptg CMHPG conc.**

The 20 wt. % was the upper limit concentration conducted in this research due to the fact that the GLDA is a liquid containing about 40% of active chelating agents diluted in water. Mixing more

than 20 wt. % of chelating agents in the system will require more than half of the solution to be GLDA, which in industrial applications, is considered economically unviable.

In another experiment, a 10 wt. % GLDA effect was observed where it shows a strong water-softening effect on the 70k ppm TDS PW as well as a noticeable thickening behavior with a 50 lb/1000 gals of CMHPG polymer. The test was performed under 200°F, 500 psi and 100 $s^{-1}$ shear rate. Under elevated temperatures, also the stability is shown to be different with GLDA, as highlighted in the literature to be withstanding up to 400°F temperatures. In **Fig. 17**, the solution containing 10 wt. % GLDA stayed stable at around 73 cP for 25 minutes while the polymer alone solution degraded to 64 cP under the same time.

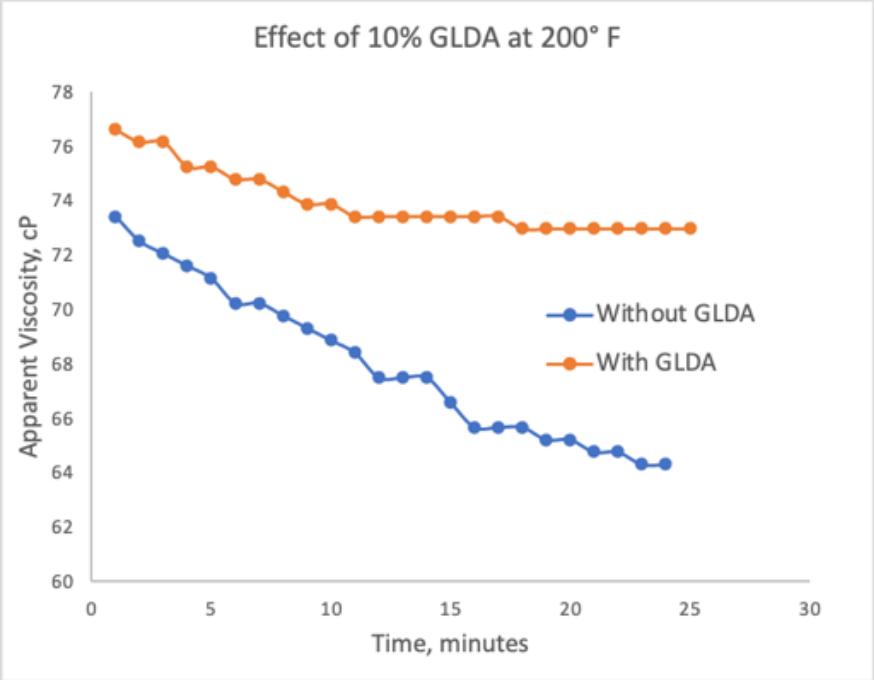


**Figure 17: Viscosity measurement over time for samples with and without GLDA under 200°F, 100 shear rate and 8 pH.**

## 4.5 CMHPG Concentration Variation:

In this experimentation, increasing only the CMHPG polymer concentration is studied in PW solutions containing 10 wt. % GLDA. The temperature was fixed at 150°F, the pressure at 500 psi and shear rate at 100 $s^{-1}$. The pH system in this experiment was also held constant at 7.5. Three concentrations of CMHPG were tested, 30, 40 and 50 lb/1000 gals. The results in **Fig. 18** showed that 50 lb/1000 gals, the highest CMHPG concentration resulted in the highest apparent viscosity, with 112 cP. 40 lb/1000 gals resulted in apparent viscosity of 73 cP while the 30 lb/1000 gals concentration resulted in 33 cP, indicating that polymer loading directly enhances the thickening behavior in such solutions.

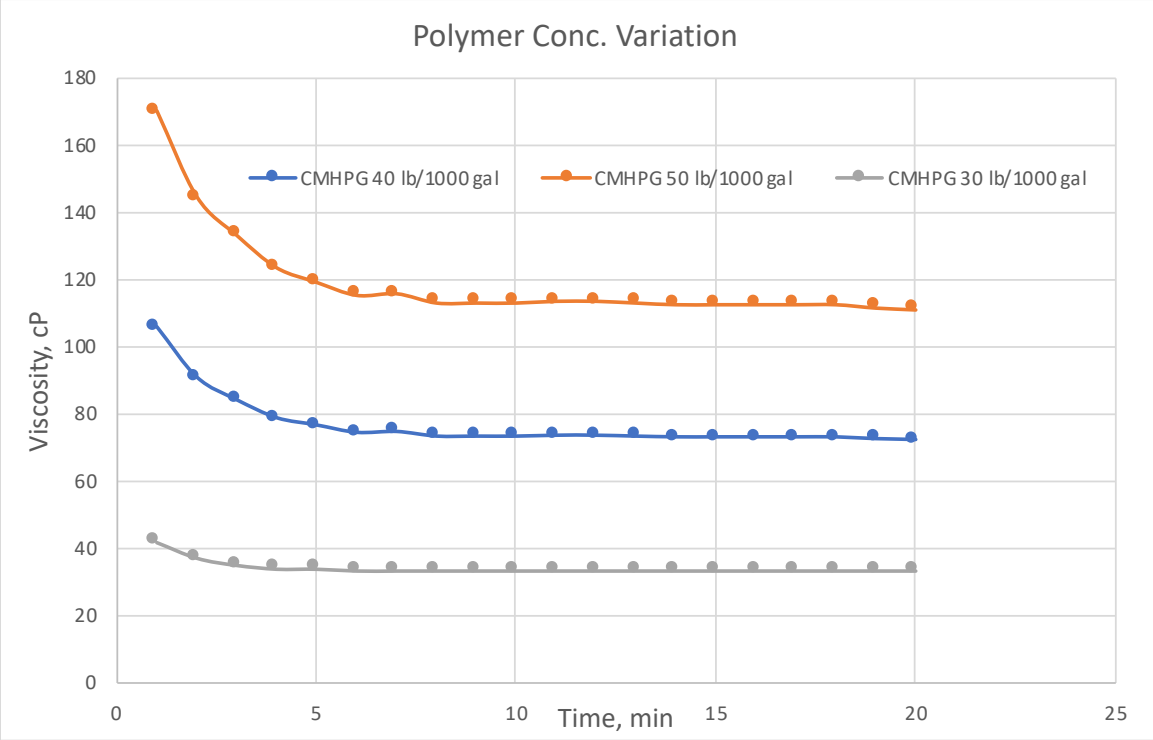


**Figure 18: Viscosity measurement over time for multiple polymer concentrations under 100 shear rate, 150 F and 8 pH.**

## 4.6 stability and Breakage Behaviors:

The stability of the newly developed stimulation fluid is highly dependent on the temperature, polymer and chelating agent concentrations. In a 10 wt. % GLDA, 50 lb/1000 gals CMHPG polymer concentrations and under 150°F temperature, the fluid stabilized for around 2 hours and broke completely after total time of 4.5 hours, shown in **Fig. 19**.

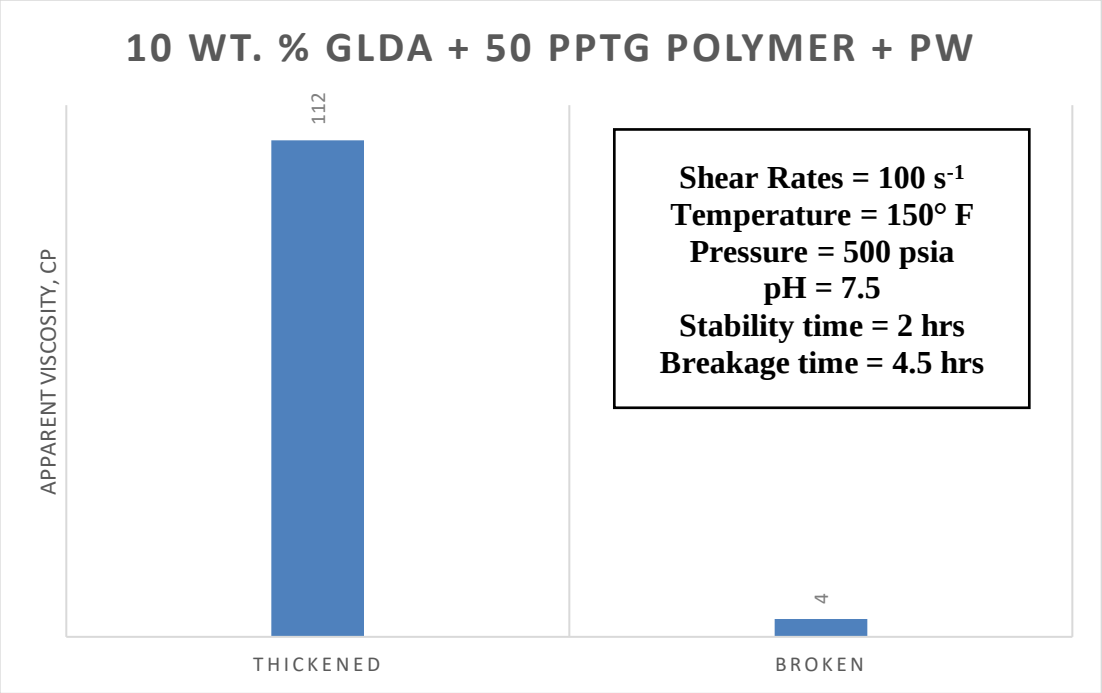


**Figure 19: Thickening and breaking behaviors with respective parameters**

In a 20 wt. % GLDA, 50 lb/1000 gals CMHPG polymer concentrations and under 150°F temperature, the fluid stabilized for around 2.4 hours and broke completely after total time of 6 hours, however reading higher viscosity values as indicated in **Fig. 20**.

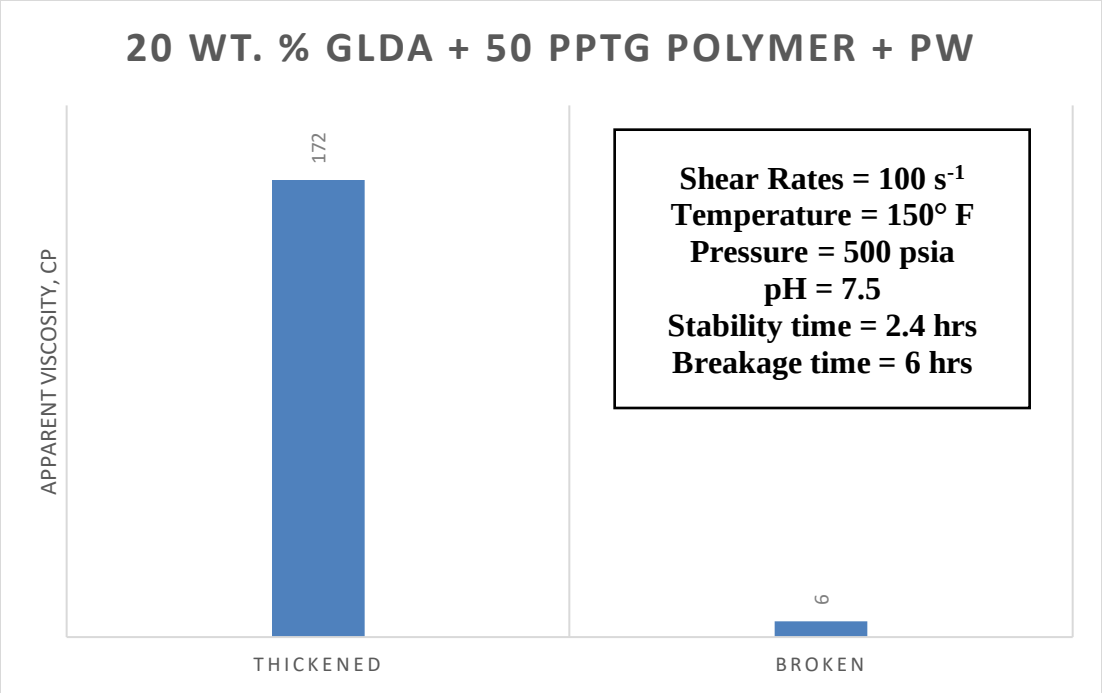


**Figure 20: Thickening and breaking behaviors with respective parameters**

However, elevating the temperature while holding the remaining of the parameters constant, clearly showed a variety of stability profiles, as shown in **Fig. 21**, in a 10 wt. % GLDA, 50 lb/1000 gals CMHPG polymer concentrations and under 200°F temperature, the fluid stabilized for around 1.5 hours and broke completely after total time of 4 hours, while also showing less apparent viscosity, 73 cP.

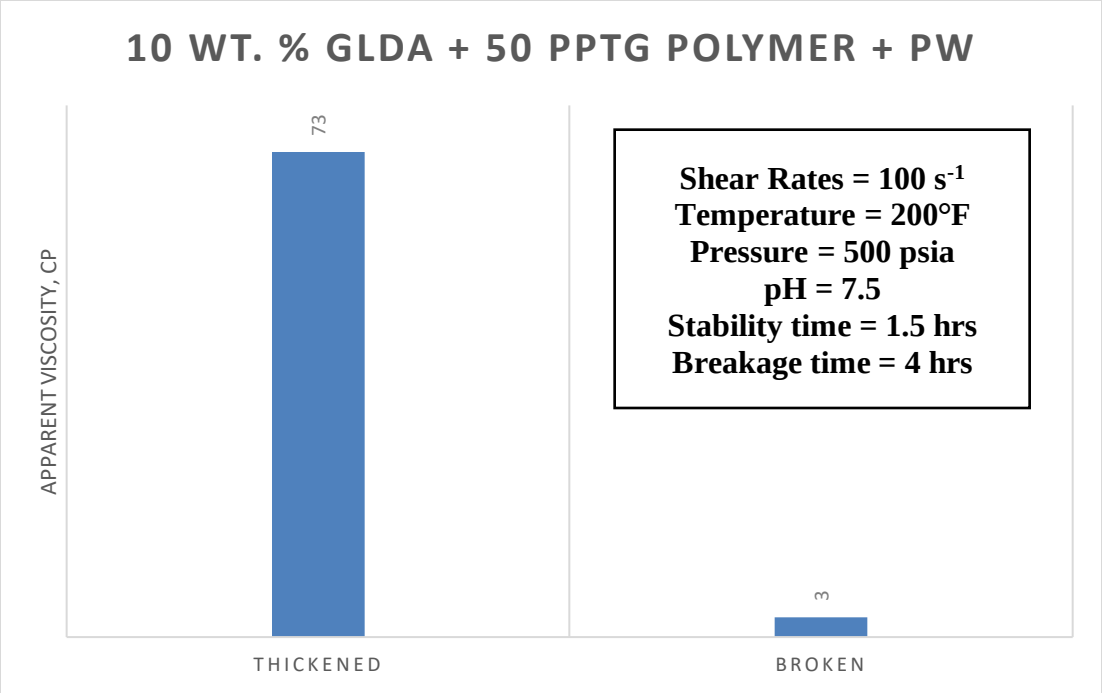


**Figure 21: Thickening and breaking behaviors with respective parameters**

Finally, setting the experiment at the highest temperature parameter, at 300°F, have drastically changed the stability time window. In a 10 wt. % GLDA, 50 lb/1000 gals CMHPG polymer concentrations, the fluid stabilized for only 0.5 hours and broke completely after total time of 1.0 hours, showing an apparent viscosity of 44 cP at thickening stage. **Fig. 22**. The before and after breaking was also physically apparent, more so in this elevated temperature of 300°F, rather than 200°F or 150°F. As seen in the **Fig. 23**, the color as well as the rheology are drastically different after a 2 hours test.

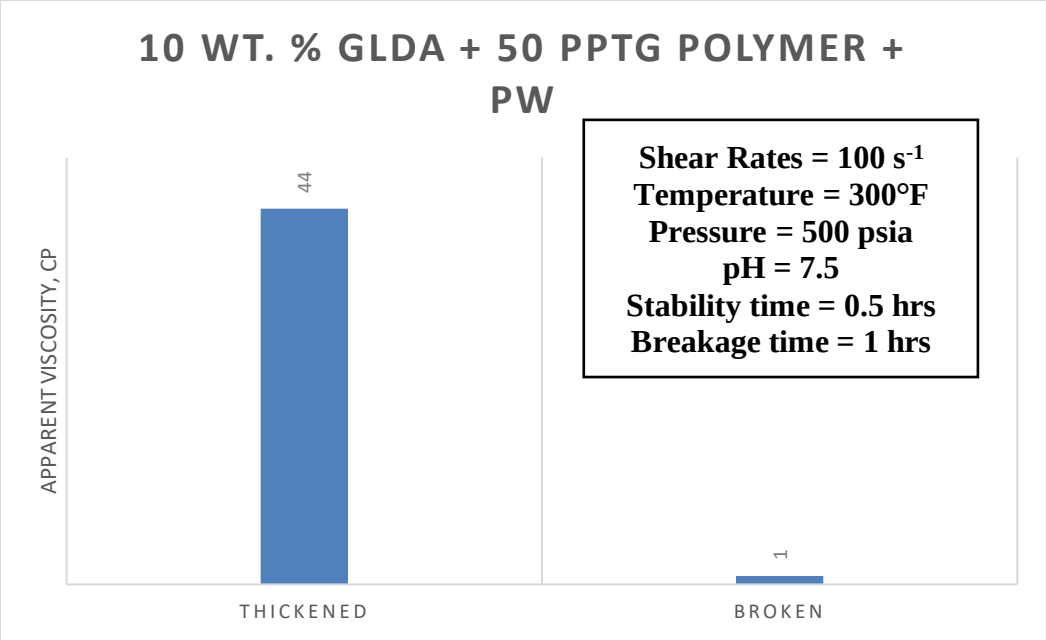


**Figure 22: Thickening and breaking behaviors with respective parameters**


**Figure 23: Broken fluid at 300°F on the left and prepared fluid before testing under HPHT on the right**

It was also noticed that introducing an oxygen scavenger, known as gel stabilizer in industrial applications, is seen to prolong the stability of the fluid at elevated temperature and delaying the breaking. An addition of 14 gpt of gel stabilizer has extended the breakage behavior in a 10 wt. % GLDA, 50 lb/1000 gals CMHPG polymer concentrations and under 200 F temperature by about 30% hours. In **Fig. 24**, a broken fluid is shown after 4 hours' test under 200 F containing gel stabilizer.

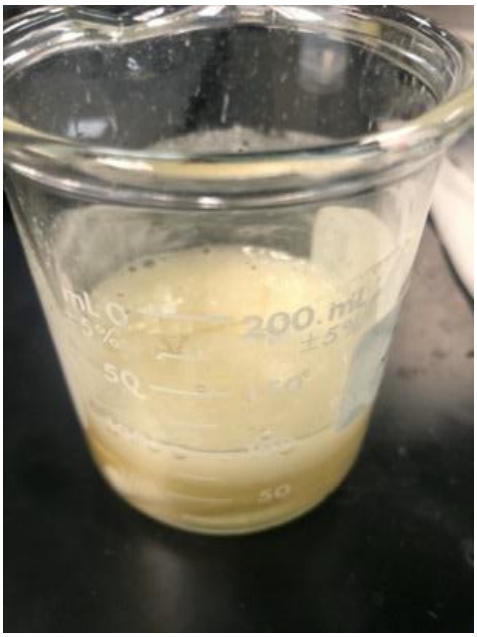


**Figure 24: Broken fluid at 150 F**

# CHAPTER 5

# CONCLUSION AND RECOMMENDATIONS

## 5.1 Conclusion

In this study, a new environment-friendly stimulation fluid is developed aiming to alleviate the burden on the exhausted groundwater resources in the kingdom, usually availed to support well stimulation jobs. The new simply-constructed fluid is based on oilfield produced water, introduced a re-use opportunity potentially eliminating environmental impacts associated with disposing and discharging such wastewater streams, promoting a more sustainable water use practice across a vital industry such as the petroleum industry. The newly developed fluid is comprised of only chelating agents GLDA, polymeric gel CMHPG and PW as base fluid, whereas in comparison to conventional stimulation fluids, several more additives are used to meet the rheological requirements for stimulation fluids.

A rheological characterization was conducted on this newly developed fluid, studying multiple parameters effect such as concentrations of polymer, concentration of chelating agent, pH, and temperature. Viscosity profiled against time were investigated while also assessing the thickening and breakage profiles of these fluids at different concentrations and settings.

The research showed that GLDA has an excellent water-softening effect as well as thickening effect when mixed with CMHPG polymer, as well as an ability to break by itself without adding a breaker agent. The study showed that the most optimum concentrations for GLDA and CMHPG

are 20 wt. % and 50 pptg respectively, while the most optimum conditions were neutral pH system of 7.5, and temperature of 150 F.

The highest apparent viscosity profile, using mentioned optimum concentrations and conditions, was at 172 cP under 100 $s^{-1}$ shear rate, exhibiting a stable thickened phase for nearly 150 minutes before breaking completely in a total of 360 minutes.

The findings of this research can aid researchers in the oil and gas upstream business to search for new ways to develop stimulation fluids and find alternatives to using freshwater resources in stimulation. The concentrations and conditions and therefore the viscosity profiles will highly depend on the intended specific application targeted.

## 5.2 Recommendations

- 4 wt. % GLDA offers great water-softening capabilities in holding off major divalent ions present in PW, with up to 10k ppm hardness, this can be utilized in multiple applications to prevent scaling.
- Neutral pH system with 20 wt. % GLDA concentration and 50 pptg polymer conc. results in adequate viscosity values for fracturing fluids carrying proppant such as hydraulic fracturing applications.
- The low pH with 20 wt. % GLDA concentration and 50 pptg polymer concentration can be utilized in acid fracturing applications.
- High pH GLDA offers a unique behavior in slowly building up viscosity profiles (5-6 hours), this can be further investigated for other applications such as water shut-off.

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