Summer 2022

Evaluation of recrosslinkable preformed particle gel (RPPG) as a fluid loss control material during drilling operations

Mohamed Saad M. Ahdaya

Department: Geosciences and Geological and Petroleum Engineering

EVALUATION OF RECROSSLINKABLE PREFORMED PARTICLE GEL (RPPG) AS A FLUID LOSS CONTROL MATERIAL DURING DRILLING OPERATIONS

by

MOHAMED SAAD M. AHDAYA

A DISSERTATION

Presented to the Graduate Faculty of the

MISSOURI UNIVERSITY OF SCIENCE AND TECHNOLOGY

In Partial Fulfillment of the Requirements for the Degree

DOCTOR OF PHILOSOPHY

in

PETROLEUM ENGINEERING

2022

Approved by:

Dr. Baojun Bai, Advisor
Dr. Thomas Schuman, Co-Advisor
Dr. Ralph Flori
Dr. Hongyan Ma
Dr. Ahmed Algarhy Mohamed

PUBLICATION DISSERTATION OPTION

This dissertation consists of the following four articles, formatted in the style used by the Missouri University of Science and Technology:

Paper I, found on pages 10–35, has been published in the *SPE Journal*, March 2022.

Paper II, found on pages 36–62, has been submitted to *Journal of Petroleum Science and Engineerin*g.

Paper III, found on pages 63–84, is intended for submission to *Journal of Petroleum Science and Engineerin*g.

Paper IV, found on pages 85–107, is intended for submission to *Journal of Petroleum Science and Engineerin*g.

ABSTRACT

One of the most intense, expensive, and time-consuming problems during drilling operations is the loss of circulation, which could result in several consequences, including wellbore collapse, fluid inflow, formation damage, environmental issues, and nonproductive time. This research aims to evaluate novel materials that can mitigate lost circulation and overcome the limitation of other materials. In this research, a comprehensive evaluation of a re-crosslinkable preformed particle gel (RPPG) has been conducted to determine the extent to which it can be used to control drilling fluid losses during drilling operations. The RPPG consists of swellable gel particles that can self-crosslink to form a strong bulk gel in fractures to form strong plugging after being placed in the loss zones. Different RPPGs were investigated and evaluated for different reservoir temperature conditions, including Low-Temperature RPPG for reservoirs up to 80°C, Medium Temperature RPPG (80 to 100°C), and High-Temperature RPPG (130 °C). The effect of the RPPG swelling ratio, drilling fluid, conventional LCMs, and fracture width on its plugging efficiency were investigated. Based on this research, the LT-RPPG can withstand pressure up to 1381 psi/ft in a 2.0 mm fracture width, while MT-RPPG can seal fracture widths up to 3.0 mm and hold pressure up to 6234 psi/ft when a swelling ratio of eight is used. The HT-RPPG sealing pressure can reach up to 1077 psi/ft when a fracture width of 1.5 mm is used. Hence, Each RPPG achieved an excellent plugging and sealing performance related to its application. Thus, the RPPGs can be an excellent candidate to work as a fluid loss control material during drilling operations.

ACKNOWLEDGMENTS

IN THE NAME OF ALLAH, THE MOST BENEFICENT, THE MOST MERCIFUL.

I would like to thank my advisor, Dr. Baojun Bai, for all the support, help, and guidance he gave me during my Ph.D. He was an incredible advisor and an incredible listener. He constantly helped me put things into perspective and prepare me for great things in the future. I cannot express enough my gratitude.

I would like to thank my co-advisor, Dr. Thomas Schuman, for his patience, motivation, guidance, and support.

I would like to express my gratitude to my committee members, Dr. Ralph Flori, Dr. Hongyan Ma, and Dr. Ahmed Algarhy, for their help and valuable feedback.

I would like to thank my father, mother, and four sisters for their support and encouragement.

I would like to acknowledge my research group members, especially Ali Al Brahim, for their great help and valuable discussions.

I would like to thank my wife, Arwa, and my three daughters, Rawan, Lujain, and Yasmine, for their constant support and motivation. I am really grateful for their support which helped a lot during my study.

TABLE OF CONTENTS

Page

LIST OF ILLUSTRATIONS

LIST OF TABLES

NOMENCLATURE

Symbol	Description
RPPG	Re-Assembling Preformed Particle Gel
KCl	Potassium Chloride
SEM	Scanning Electron Microscope
Frr	Residual Resistance Factor
HPAM	Hydrolyzed Polyacrylamide Polymer
PEI	Polyethleneimine
GP-A	Hydrophobic Association Supramolecular Hydrogel
ECS-1	Engineered Composite Solutions
DPP	Deformable Plugging Polymer
AMPS	2-Acrylamide-2-Methylpropionic Sulfonic Acid
SPAM	High-Temperature Resistance Acrylamide Based Polymer
SR	Swelling ratio

1. INTRODUCTION

1.1. STATEMENT OF PROBLEM

1.1.1. Lost Circulation Overview. One of the common problems during drilling operations is lost circulation which are mostly encountered when drilling into highly permeable formations, depleted reservoirs, and fractured or cavernous formations. Usually, when the lost circulation begins in the fractured formation, the fractures extend due to the drilling fluid (Moore, 1986). The drilling fluid losses can be classified into seepage, partial, severe, and complete (total) losses based on the volumes of mud losses (Nayberg & Petty, 1986). Seepage losses are when the volume of the losses reach up to 10 bbl/hr. When the losses are higher than 10 bbl/hr and up to 20 bbl/hr, the losses are considered partial losses. The severe losses are encountered when the losses exceed 200 bbl/hr. However, the complete loss is when no drilling fluids return to the surface.

1.1.2. Lost Circulation Consequences. Lost circulation has many consequences such as increasing nonproductive time (NPT) and loss of the drilling fluids into the formation, resulting in an increase in the total cost of the drilling operations (Howard and Scott 1951; Kumar et al., 2010). Moderate to severe loss circulation problems that lead to more NPT results in a significant increase in the total operational cost besides the cost of drilling mud, lost circulation materials (LCMs), and the treatment. Al-Arfaj, et al. (2018) highlighted that 10% of the nonproductive time in the Gulf of Mexico, based on the 1993 to 2003 drilling history, was a result of different types of loss circulation problems. This 10% increase can result in an enormous increase in the total cost of the drilling operation, because of the additional rental costs that would be required, especially for offshore rigs

which cost much higher than the onshore rigs. According to Cook et al., (2011), Sanders, et al. (2010) and Al Maskary, et al. (2014) the worldwide estimation cost of lost circulation problems are 1 to 4 billion dollars annually. It could be a very costly drilling problem if not being prevented or controlled immediately after its occurrence. Kumar and Savari (2011) mentioned that a series of other drilling problems could occur even if only one lost circulation was encountered, adding around a million dollar or more per incident.

The lost circulation problems are not limited to one area. These problems can be encountered at any depth when the pressure applied to the formation exceeds the formation breakdown pressure. The loss of circulation could lead to the loss of well control which could cause blowouts. This may occur due to the decrease in the wellbore's hydrostatic pressure as a result of losing drilling fluids into the formation. Maintaining the wellbore's hydrostatic pressure is one of the drilling fluids functions. Reduction in hydrostatic pressure could cause a kick, which is an entry of formation fluids, such as gases and hydrocarbons, into the wellbore. Kicks may expose the crews to risky situations.

1.1.3. Lost Circulation Treatment. Lost circulation treatment can be categorized based on the period in which they were introduced to two categories, preventive and corrective.

1.1.3.1. Preventive treatment. Before entering the thief zone, any treatment that is applied to prevent or reduce the losses is known as a preventive treatment. Wellbore strengthening, which is achieved by sealing the fractures that enhances the fracture gradient and widen the operational window, is the objective of using the preventive methods (Whitfill, and Miller, 2008; Salehi and Nygaard, 2012). Wellbore strengthening enhances the efficient pressure of the crack and expands the mud-weight range. The wellbore

strengthening was generally assumed to occur by bridging, trying to plug, or cure cracks of which mud loss arises (Feng, et al., 2016). In addition, to avoiding or alleviating the circulation loss, wellbore strategies for improving the system may also reduce the number of non-productive times involved, such as wellbore dysfunction, pipe trapping underwater blowout, and kickback.

1.1.3.2. Corrective treatment. Any method that is applied after the fluid loss occurs is known as a corrective treatment (Kumar and Savari, 2011). In this method, the lost circulation materials are introduced to the drilling fluid directly or it can be spotted as a concentrated pill (Alsaba, 2015). Lost circulation materials (LCM) are the materials that are usually added to the drilling fluids to seal the formation fractures or reduce the formation permeability to cure the losses by increasing the amount and the size of the particles inside the mud (White, 1956). LCMs may be dispersed in the active system or mixed in a high concentration and placed against the thief zones referred to as LCM pills (Kefi et al. 2010; Almagro et al. 2014, Jeennakorn, 2017).

The overall goal of wellbore remedies is to stop fracture propagation and enhance the pressure that a wellbore can withstand without substantial fluid losses. One or a combination of the two strategies for remediation is supposed to improve the pressure carrying capacity in the wellbore. Different research reports have been published on wellbore remedial consolidation. The efficacy of the remedial reinforcement treatments is often examined by repeated leak-off tests. Repetitive leak-off experiments can be performed by injecting liquids with and without LCM to determine the crack pressure for the enhanced and un-reinforced borehole. Both lab studies and field jobs have already shown that fracture stress can be significantly enhanced (Benyeogor, et al., 2016).

1.1.4. Lost Circulation Materials. Conventional Lost Circulation Materials (LCM), which is usually used to mitigate seepage or partial losses, is small particles suspended in the drilling fluid. Their function is to plug the minor fractures in the formation to prevent losses into the formation. Table 1.1. shows the lost circulation materials and their limitations. The classification of conventional LCM usually depends on its appearance or physical properties. These classifications are fibrous, flaky, and granular or a blend of the three (Howard and Scott, 1951; White, 1956; Canson, 1985). Since LCM materials have different physical and chemical properties and their applications, Alsaba et al. (2014) reclassified them into seven categories including granular, flaky, fibrous, a mixture of LCMs, acid-soluble/water-soluble, high-fluid-loss LCMs squeeze, swellable/hydratable LCM combinations, and nanoparticles.

Evaluating the performance of LCM treatments has been investigated using different methods. Measuring the volume of fluid loss using constant pressure is one of these methods. Usually, these experiments are conducted using high pressure high temperature (HPHT) filter press or particle plugging apparatus (PPA) with slotted disks, which represent the fractured formations (Whitfill, and Miller, 2008; Kumar, and Savari, 2011). Another method that has been used is to evaluate the sealing efficiency of the LCM is by using fractured cores (Hettema, et al. 2007; Sanders, et al., 2008; Van Oort, et al., 2011). Several researchers addressed the issue of fluid losses during drilling operations. Even though many plugging materials such as foams, cement, suspended particles, and gelled polymers have been investigated, the lost circulation problems are still challenging (Fitch, and Minter, 1976; Du, et al., 2017). Thus, studying the potential of using new materials is highly recommended to prevent losses during the drilling operations.

Table 1.1. Lost circulation materials and their limitations

LCM	Examples	How it works	Limitations
Fibrous	Polypropylene fiber Cedar wood fibers Nylon fibers	It forms a mat-like bridge over porous formations. These mat effects a reduction in the size of the openings to the formation.	Size gradation obtainable Resistance to disintegration Degradation when circulated in the mud system
Flaky	Mica Cellophane flakes Cottonseed hulls	Designed to bridge and to form a mat on the formation face. Can plug and bridge many types of porous formations to stop the mud loss or to establish an effective seal over many permeable formations.	Size and strength Large fractures
Granular	Walnut shells Gilsonite Asphalt	It forms bridges either at the formation face or within the formation matrix They are available in a wide particle size distribution	Its effectiveness depends primarily on proper particle-size distribution to build a bridge
Mixture	Blends of two or more of the conventional LCM	This blend will penetrate fractures and seal them off more effectively Using blend of various LCMs provides advantage of having different particle size and types of materials to provide effective sealing	Fail to seal large fracturs
Slurries /Settable	Cement Diesel oil- bentonite-mud mixes	Usually used when no return. They designed to get harden with time.	Time consuming for rig Affected by mud contaminations

In this research, novel materials, known as recrosslinked preformed particle gel (RPPG), will be introduced. RPPG is a swellable polymer gel that can swell the brine,

drilling fluid, or formation water to form a bulk gel at loss zones to plug or seal any type of fractures. Several researchers studied the RPPG for conformance control application, and based on their findings, RPPG showed excellent performance (Pu et al., 2019; Long et al., 2020; Song, et al., 2022a; Song et al., 2022b; Ahdaya et al., 2022). RPPGs are non-toxic and environmentally friendly materials that can be used as fluid loss control agents. RPPG crosslinked time can be easily controlled, which will significantly contribute to reducing the waiting time based on the application and the circumstances in each situation. RPPG strength is strongly dependent on the swelling ratio and can be controlled by increasing or decreasing the RPPG swelling ratio. Based on these features, the RPPG is an excellent candidate for controlling severe losses during drilling through loss zones.

1.2. EXPECTED IMPACTS AND CONTRIBUTION

Different types of RPPG were evaluated in this research to study their capability to control the losses especially the severe and total loss that cannot be easily controlled. The succusses of this research will promote the use of RPPG to enhance the wellbore strengthening and curing the severe loss during the drilling operations. A summary of the benefits of using the RPPG is listed below:

- The ability to control the RPPG swelling ratios and strength will aid in being used in different formations and conditions.

- Controllable re-crosslinkable time material can be very beneficial for field application as it can significantly reduce the nonproductive time.

- Using Low-temperature RPPG could control the severe losses at low-temperature reservoirs, up to 80 °C, and seal the fractured formation.
- Medium-temperature and high-temperature RPPG are designed to control severe losses in high-temperature environments (80 to 130 °C).
- Mixing RPPGs with different conventional materials could enhance their plugging and sealing efficiency and results in more robust materials that can withstand harsh environment.

1.3. OBJECTIVE

The main objective of this research is to evaluate different types of RPPG, including low temperature, medium temperature, and high temperature RPPGs, and examine its ability to reduce or mitigate the severe fluid losses during drilling operations. The following objectives will be achieved from this research:

- A study of the capability of using the RPPGs to control the fluid loss.
- An investigation of the effect of the RPPG swelling ratios on the strength of the material and its plugging efficiency.
- An examination the effect of waiting time on the RPPG sealing pressure.
- An investigation of the impact of the fracture size on the RPPG plugging ability.
- A study of the effect of drilling fluids on the RPPG strength and sealing efficiency.

- An evaluation of the influence of various additives on the plugging efficiency and the strength of the RPPGs.

1.4. SCOPE OF WORK

This research is divided to three different tasks based on the material to be used, each task is divided to four different subtasks. Figure 1.1 shows the scope of work for this research. The first task is evaluating the LT-RPPG (Low temperature < 80°C) as a lost circulation material for low temperature reservoirs. The task is divided to four subtasks including investigate the LT-RPPG without any additives, study the impact of drilling fluid, study the impact of adding mica, study the influence of adding walnut shell, and investigate the effect of adding fiber on LT-RPPG strength and plugging efficiency. The second and the third tasks are the same to the first task just using different materials, which are Medium Temperature RPPG (80 to 100 °C) and High Temperature RPPG (130°C) respectively.

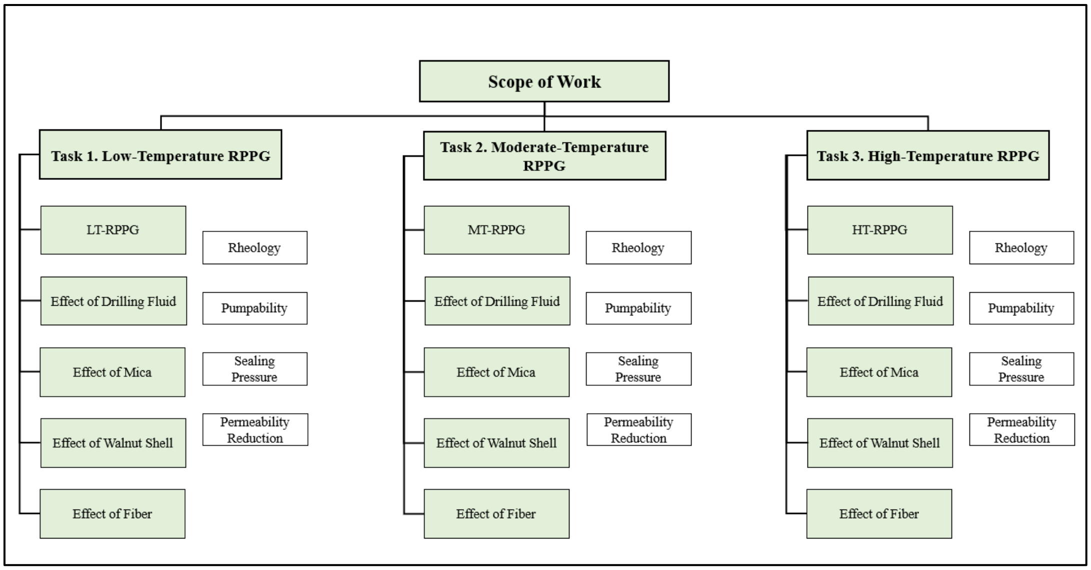

Figure 1.1. Scope of work

PAPER

I. LOW-TEMPERATURE RECROSSLINKABLE PREFORMED PARTICLE GEL AS A MATERIAL FOR LOST CIRCULATION CONTROL

ABSTRACT

One of the most prevalent, expensive, and time-consuming problems during drilling operations is the loss of circulation. Uncontrolled lost circulation of drilling fluids may lead to dangerous well control difficulties and, in some cases, complete loss of the well. In this paper, the ability of a Low-Temperature Recrosslinkable Preformed Particle Gel (LT-RPPG) has been evaluated to determine the extent to which it can be used to control drilling fluid losses during drilling operations. The RPPG consists of swellable gel particles that can self-crosslink to form a strong bulk gel in fractures to form strong plugging after being placed in the loss zones. We investigated the effect of the LT-RPPG swelling ratio and fracture width on its plugging efficiency to fractures through core flooding tests. Results showed that its sealing pressure can reach up to 1381 psi/ft and permeability reduction more than 99.99% when the RPPG swelling ratio is five for the fracture with a width of 2.00 mm. LT-RPPG is a good candidate that can be used to control the severe or total loss during drilling operations.

1. INTRODUCTION

One of the problems during drilling operations is lost circulation, which is a common phenomenon that occurs during many drilling processes. It is mostly encountered when drilling into highly permeable formations, depleted reservoirs, and fractured or cavernous formations. Usually, in shallow, unconsolidated formations, when the lost circulation begins in the fractured formation, the fractures extend due to the drilling fluid (Moore, 1986). Based on returns, the drilling fluid losses can be classified into seepage, partial, severe, and complete (total) losses when full loss of returns occurs (Nayberg & Petty, 1986). Lost circulation has many consequences, such as increasing nonproductive time (NPT), resulting in an increase in the total cost of drilling operations (Howard and Scott, 1951; Kumar et al., 2010). This loss of circulation could lead to loss of well control which could cause blowouts. According to Cook et al., (2011), Sanders et al. (2010), and Al Maskary et al. (2014), the worldwide estimated cost of lost circulation problems is one to four billion dollars annually.

Lost circulation materials (LCM) are usually added to drilling fluids to seal the formation fractures or reduce the formation permeability. Their function is to plug the minor fractures in the formation to prevent losses within the formation. They are applied to mitigate the losses dispersed in the active system or are mixed in a high concentration and spotted against the thief zones, referred to as LCM pills (Kefi et al., 2010; Almagro et al., 2014; Jeennakorn, 2017).

The classification of conventional LCM usually depends on its appearance or physical properties. These classifications are fibrous, flaky, and granular or a blend of the

three (Howard and Scott, 1951; White, 1956; Canson, 1985). Since LCM materials have different physical and chemical properties and their applications, Alsaba et al. (2014) re-classified them into seven categories including granular, flaky, fibrous, a mixture of LCMs, acid-soluble/water-soluble, high-fluid-loss LCMs squeeze, swellable/hydratable LCM combinations, and nanoparticles.

Hydratable/swellable materials are activated when in contact with chemical reagents, drilling fluids, or formation water to perform a plug to seal the thief formations. These materials can seal various fracture sizes due to their swelling capacity. Cross-linked polymer gel has been broadly used to plug severe lost circulation (Jiang, et al., 2019). Hashmat et al. (2016) studied HPAM/PEI gel using a transparent apparatus to prevent drilling fluid loss. They filled a transparent tube with glass beads of different sizes to simulate porous media. They found that HPAM/PEI gel had successfully prevented mud loss in a zone with a porosity of 36% and a permeability of 300 Darcy at room temperature and differential pressure of 150 psi. Gamage et al. (2014) studied fluid loss pills for high-temperature applications up to 140 °C. This fluid loss pill consists of a water-soluble polymer and a metal-based crosslinker; however, the gelation time was one hour, which limits its application. Jia et al. (2017) studied a polymer gel pill which consists of SPAM and PEI as crosslinker. Their finding was that the mature polymer gel could act as a fluid loss pill to withstand a high pressure of 2900 psi during overbalanced well workover and completion.

Wang et al. (2021) investigated the effect of a crosslinking agent on a supramolecular gel; using hydrophobic association polymer along with organic chromium resulted in plugging for up to 154.2 kPa (22.36 psi) when 1.2 mm fracture was used.

Magzoub et el. (2021) studied the gelation kinetics of the PAM/PEI cross-linkable polymer. They used the high-pressure, high-temperature permeability plugging apparatus. Their results showed that the sealing pressure was 400 psi with mud loss of around 200 cc when the waiting time was zero. However, it reached 2000 psi with about 40 cc of mud loss when the waiting time was 120 minutes. Cross-linked polymer gel has several limitations that can limit its application in reducing losses, such as low plugging efficiency to fracture and large openings, extremely toxic to aquatic life and carcinogenic, particularly the gels containing formaldehyde and phenol (Wang, et al., 2018; Song, et al., 2021). Gang et al. (2020) investigated a Deformable Plugging Polymer (DPP). They used sand bed experiments to test the DPP. They concluded that the plugging pressure could reach up to 1232 psi when 15% of a DPP was used. Mansour et al. (2019) studied a shape memory polymer and found that it could mitigate losses. The limitation of the smart LCM is that it highly depends on external motivation, such as temperature, to activate, or it won't mitigate the losses. A swellable polymer was applied and succeeded in maintaining low fluid loss (less than 10 bbl/hr) in a highly loss-prone zone in one of the offshore wells in East Africa when combined with ECS-1 (Savari and Whitfill, 2019).

Evaluating the performance of LCM treatments has been investigated using different methods. Measuring the volume of fluid loss using constant pressure is one of these methods. Usually, these experiments are conducted using a high-pressure, high-temperature filter press or particle plugging apparatus with slotted disks, which represents the fractured formations (Whitfill and Miller, 2008; Kumar and Savari, 2011). Another method to evaluate the sealing efficiency of the LCM is using fractured cores (Hettema et al., 2007; Sanders et al., 2008; Van Oort et al., 2011). Bao et al. (2019) investigated the

effect of LCM type, concentration, and particle size distribution on the sealing efficiency using a long-fractured slot. They found that higher LCM concentrations resulted in a higher pressure bearing. Xu et al. (2019) and Li et al. (2020) studied the effect of particle size distribution, flow rate, and particle concentrations on sealing integrity utilizing a core flooding test with fracture cores and LCM testing apparatus, respectively. Different models were used to evaluate the lost circulation materials, including the slotted disks models; however, using fracture cores setup with actual cores to evaluate the plugging efficiency and the sealing pressure is more reliable as it simulates the fractures in the reservoir. Moreover, the thickness of the slotted disk is minimal; thus, it is mainly used to evaluate the bridging materials. However, in our case, the gel can remain in the fracture even after the breakthrough, which reduces the permeability of the target formation and reduces the losses even after the breakthrough.

A new, novel Re-Crosslinkable Preformed Particle Gel (RPPG) material was introduced in 2018 by Pu et al. (2019). The RPPG is a dry, white granular particle, consisting of a crosslinked poly (acrylamide-coacrylic acid) copolymer with a pre-embedded second crosslinker (Crosslinker II). The purpose of using two crosslinkers is that Crosslinker I produces a 3-D network structure of the gel, and Crosslinker II merely participates in the synthesis stage that can endow the gel re-crosslinking property by crosslinking polymers of the different gel particles (Pu et al., 2019). After being placed in brine, RPPG can swell, and its swelling particles can re-crosslink with a controllable time and form a bulky gel. The RPPG can withstand higher pressure compared to ordinary PPG. The RPPG is marked with low-temperature RPPG (LT-RPPG) in this paper because we wanted to differentiate it from other RPPGs that were developed for high-temperature

conditions. The LT-RPPG can be stable only when the reservoir's temperature is below 80 ºC. This product was successfully applied for controlling fluid conduit problems in the West Sak field on the North Slope of Alaska. Seventeen different treatments were conducted from 2017 to 2019, and results showed a 23% improvement over traditional PPG (Targac et al., 2020). RPPG has combined the advantages of in-situ gel systems and swellable by including a good mechanical strength and thermal stability, controllable recrosslinking time, good placement in fractures, and strong plugging efficiency to fractures (Pu et al., 2019; Pu. et al., 2021). Based on these features, the RPPG is a promising material to reduce fluid loss into loss zones.

The objective of this paper is to evaluate whether the LT-RPPG can serve as an excellent plugging agent to control severe fluid losses in relatively shallow formations through a series of core flooding tests. We have evaluated the effect of the RPPG swelling ratio and fracture width on the breakthrough/sealing pressure and plugging efficiency.

2. EXPERIMENTAL MATERIALS DESCRIPTION

RPPG. A commercial RPPG, provided by Daqing Xinwantong Technology Developing Company, is used in this study. It is mainly composed of a crosslinked poly (acrylamide-co-acrylic acid) copolymer with a pre-embedded crosslinker. Swollen RPPG with different swelling ratios of 5, 8, 10, and 16 was prepared for evaluation by adding a calculated mass of brine into a given mass of dry RPPG by using the following equation:

$$W_{brine} = (SR - 1) * Wt_{RPPG} \quad (1)$$

Where SR is the swelling ratio; W_{brine} is the mass of water in grams, and Wt_{RPPG} is the RPPG mass in grams. Figure 1 shows the RPPG dry particles (a), the initial slurry during mixing (b), and the fully swollen particles (c).

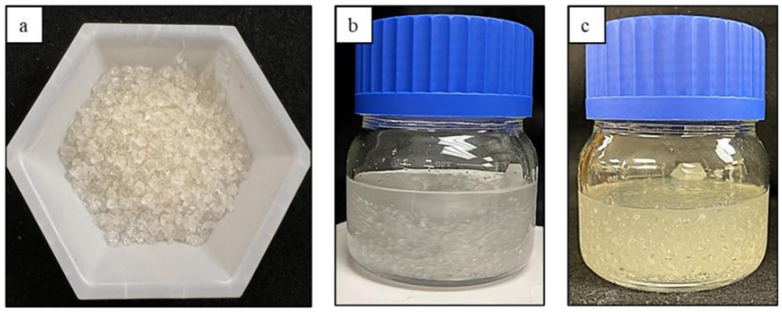

Figure 1. LT-RPPG sample (a) RPPG dry particles. (b) initial slurry. (c) fully swollen.

Brine. A solution of 2% potassium chloride (KCL) was used as a base fluid for all samples.

Cement cores. The fractured cemented cores were prepared by mixing tap water with cement powder which was then poured into the cylinder molds. A steel bar was placed in the center of the mold to create the desired fracture. The steel bar was removed 6 to 8 hours after the cement slurry was prepared and before the cement was fully set. Different fracture widths were used in this study, including 1.5 mm, 2 mm, and 3.0 mm.

HAAKE rheometer. A HAAKE rheometer was used in this study to measure the elastic modulus (G') and viscous modulus (G'') of each sample to better understand the effect of each parameter on the material's strength.

Scanning electron microscopy (SEM). The microstructure of RPPG was characterized with the Hitachi S-4700 Field Emission Scanning Electron Microscope (SEM).

3. EXPERIMENTAL SETUP DESCRIPTION

Figure 2 shows the setup design used to perform the experiments. The design consists of a core holder to hold the fractured cement core, an accumulator that contains the sample and is used to inject the sample into the fractured cement core, a pressure transducer that connects to the computer to record the pressure changes during the experiment, and a syringe pump to apply the flow rates.

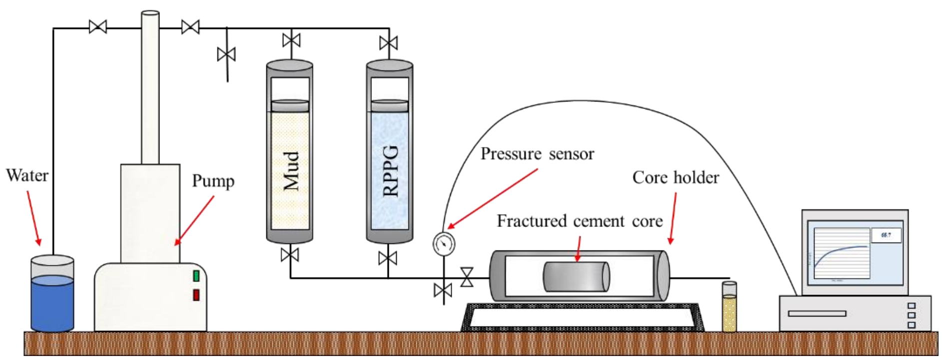

Figure 2. Core flooding setup design

4. EXPERIMENTAL PROCEDURE DESCRIPTION

In this experiment, core flooding experiments were conducted to measure the breakthrough pressure (also called sealing pressure), which is the highest pressure that the material can withstand before it cracks or builds channels within the material, and plugging efficiency, which is expressed using residual resistance factor (Frr). The fractured cement core was first placed in the core holder and then the drilling fluid was injected into the fractured core. After that, the swollen RPPG sample with the designed swelling ratio was

injected into the core using a constant flow rate of 1 ml/min until a stable pressure was reached. Then all the connecting tubings were cleaned to ensure no RPPG remained in the tubings. The fractured cement core remained in the core holder at room temperature for 24 hours for the RPPG to cure. Then the drilling fluid was injected again to measure the sealing pressure. Finally, 2% KCl brine was injected into the core to measure Frr at different flow rates of 0.10, 0.25, 0.50, 1.00, and 2.00 ml/min after the sealing pressure was obtained. Then, the permeability was calculated at each flow rate using the Darcy equation:

$$k = \frac{q\ \mu\ L}{A\ \Delta P} \quad \text{.....(2)}$$

Where K is permeability, md, Q is flow rate, cc/sec, μ is viscosity, cp, L is core length, cm, A is core cross-section's area, cm^2, and ΔP is differential pressure, atm.

Then, the Frr was calculated using the following equation:

$$F_{rr} = \frac{K_{before}}{K_{after}} \quad \text{.....(3)}$$

Where K_{before} is the permeability of the fracture before the treatment and K_{after} is the permeability of the fracture after the treatment.

5. RESULTS

5.1. EFFECT OF SWELLING RATIO ON GEL STRENGTH

Different LT-RPPG swelling ratios were investigated in this research. Table 1 shows the samples of LT-RPPG. The samples were prepared by the procedures, as follows. Initially, the brine was added to a beaker and placed on the magnetic stirrer. Then, the LT-RPPG particles were added slowly to avoid flocculation. After that, the mixture was left to

mix until the LT-RPPG particles absorbed all the water. Figure 3 shows the G' and G'' for samples 1, 2, 3, and 4. The G' and G'' results were 235, 706, 1765, and 3590 Pa for ratios 1:16, 1:10, 1:8, and 1:5, respectively. Figure 4 shows the images of sample 1 after different times. After the mixing finished and the LT-RPPG particles absorbed all the water, the sample was left for recrosslinking, and pictures were taken at different times. As shown in Figure 4, the LT-RPPG particles were still not recrosslinked after 1 hour after mixing was finished. It started recrosslinking after about 6 hours, and it became fully recrosslinked after around 24 hours. Picture (d) in Figure 4 shows the strength of the material after 72 hours. The pores' size seems to be highly compacted, resembling a honeycomb structure, as shown by red circles. Figure 5 shows the SEM image of the RPPG after fully recrosslinked. The pores are strongly interconnected, having a thick wall, as shown by blue arrows.

Table 1. LT-RPPG samples

Sample #	LT-RPPG Ratio	Brine Concentrations (%)
Sample 1	1:16	2 % KCl
Sample 2	1:10	
Sample 3	1:8	
Sample 4	1:5	

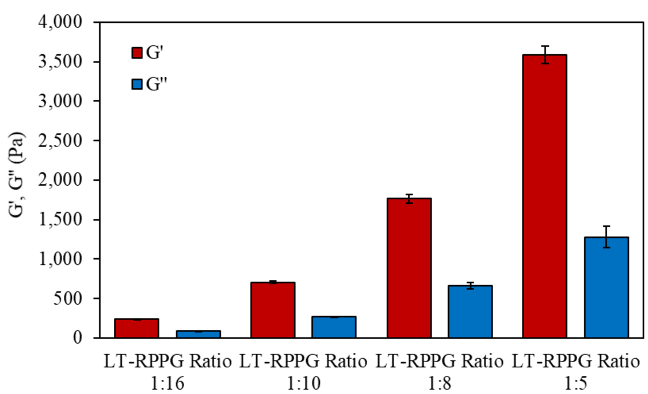

Figure 3. The G' and G" for samples 1, 2, 3, and 4

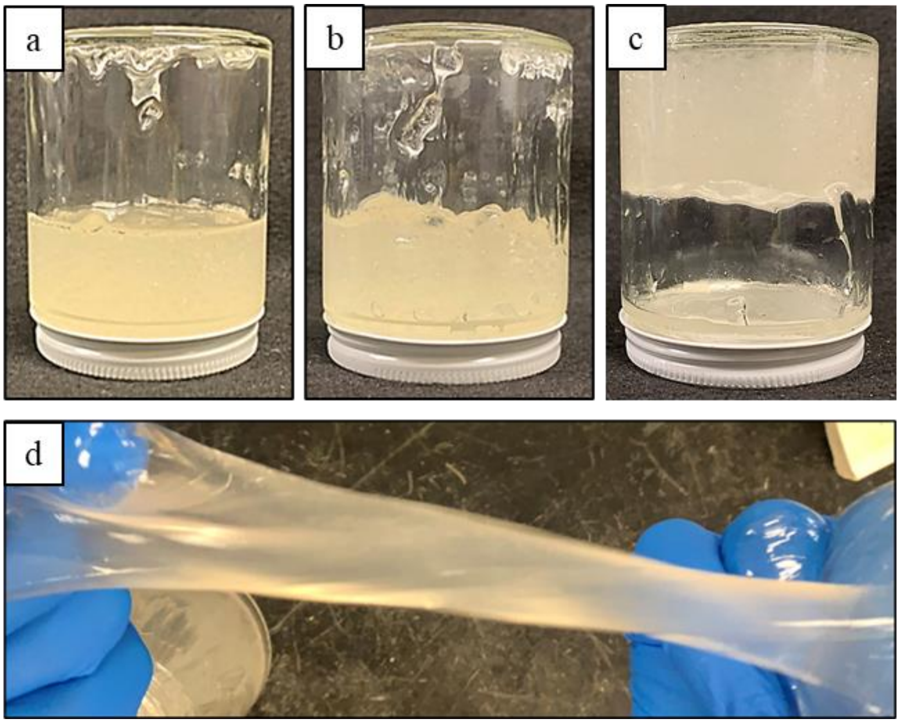

Figure 4. Pictures of sample 1 after different time. (a) immediately after mixing. (b) after 1 hour. (c) after 24 hours. (d) after 72 hours.

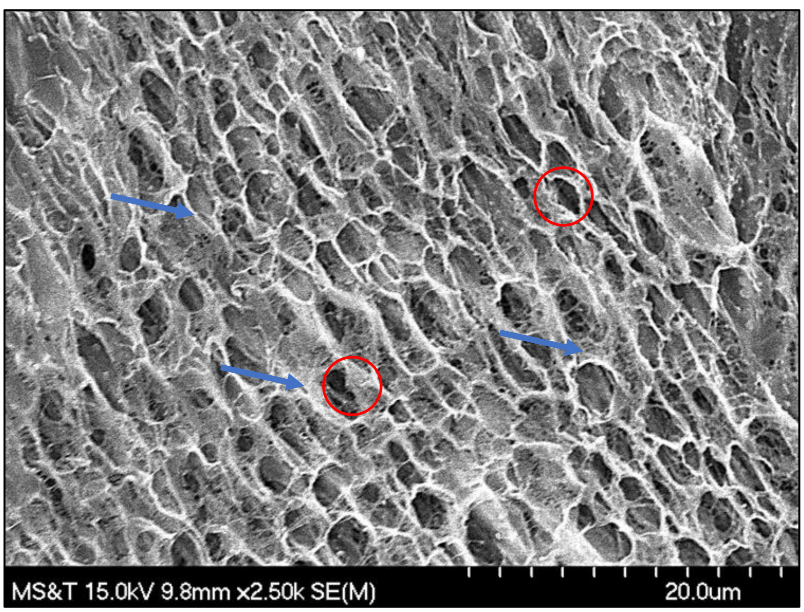

Figure 5- SEM obtained with S4700.

5.2. CORE FLOODING EXPERIMENTS RESULTS

A series of experiments was conducted to study the effect of fracture width and swelling ratios on the sealing pressure and plugging efficiency. The results of these experiments are discussed in this section.

Figure 6 shows the experiment results for the sample of LT-RPPG with a ratio of 1:10 mixed with 2% KCl brine. The experiment was conducted using a fracture width of 3.0 mm. As shown in the graph, the LT-RPPG injection pressure increased for the first 120 minutes and then stabilized at about 250 psi/ft. The core, with the sample inside it, was left for the recross-linking process for 24 hours. After that, the drilling fluid was injected using a flow rate of 1 ml/minute. The drilling fluid injection was used to measure the sealing pressure. The sealing pressure for this experiment, when 3 mm fracture width was used, reached 183 psi/ft.

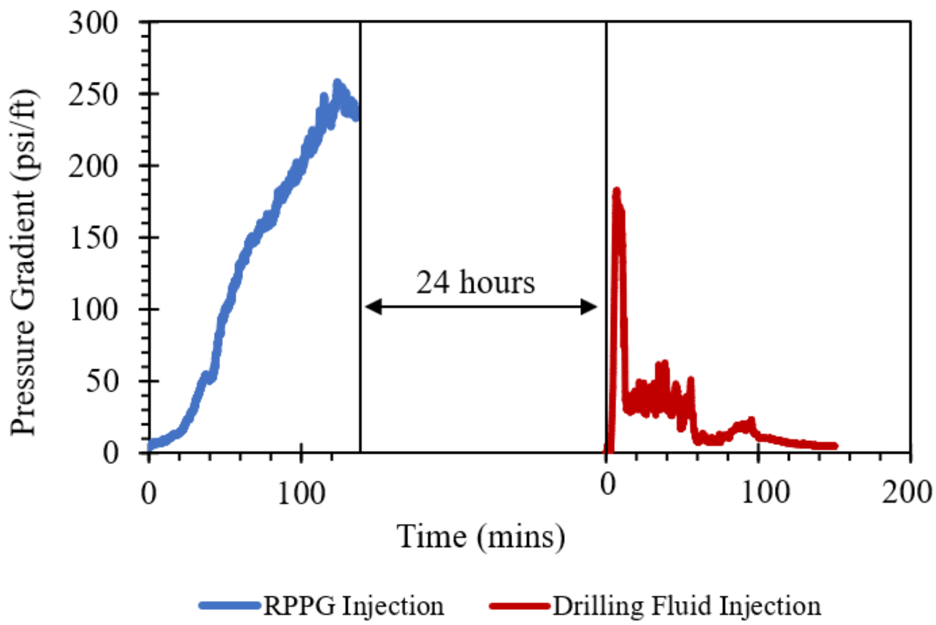

Figure 6. Sample 2 core flooding results

Figure 7 shows the fractured cement core before and after the experiment. Figure 8 shows the inside of the fracture after the experiment. As shown in the pictures, there was a residual amount of the mixture after the post-injection, which played an essential role in reducing the permeability inside the fracture. The permeability reduction for this sample was more than 99.99% as it was about 7.59 E+05 Darcy before treatment and was reduced to less than 46 md. The Frr was measured to test the plugging efficiency for this sample. Figure 9 shows the Frr results at different flow rates. The Frr decreased with increasing the flow rate. To understand the extrusion effect on gel strength when the swollen RPPG passed through the facture under pressure, the RPPG rheological properties were evaluated by collecting samples from the effluents. The results showed the G' slightly increased from 802.60 Pa to 990.88 Pa, which is caused by the slight dehydration of the swollen gel.

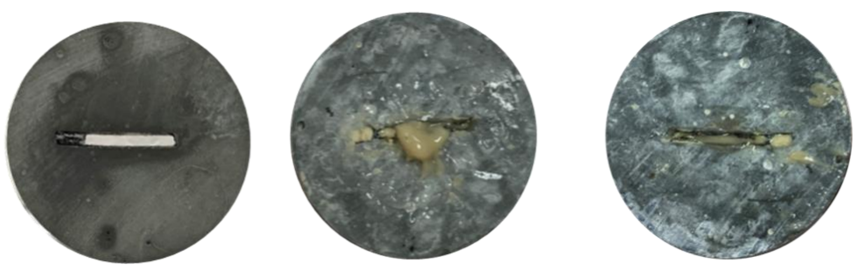

Figure 7. Fractured cement core before and after the experiment

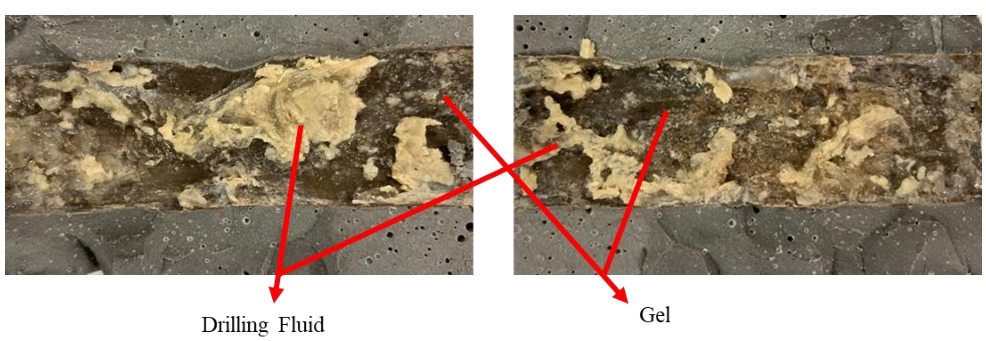

Figure 8. Image inside the fracture after the experiment

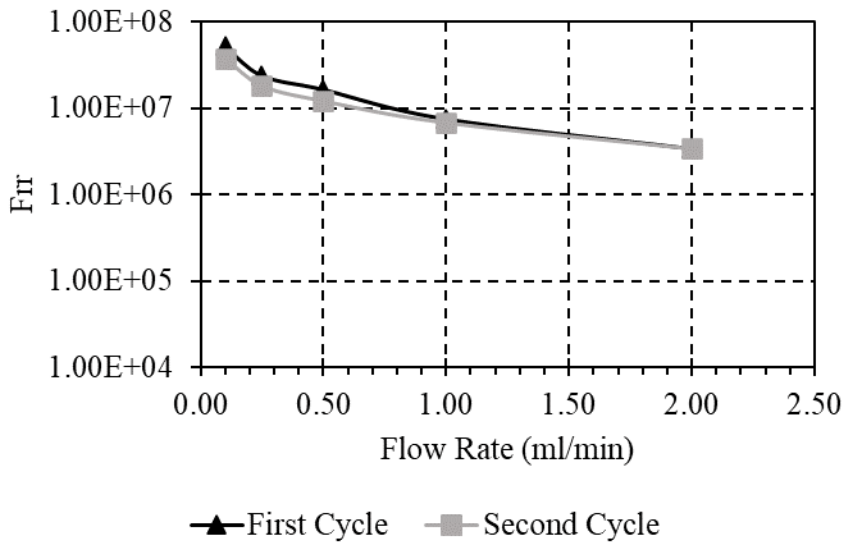

Figure 9. Frr results for sample 2 at different flow rates

5.2.1. Fracture Width Effect. Fracture width is considered one of the critical parameters that can significantly impact drilling fluid loss during drilling operations. Fractures, which naturally exist in most formations, can be induced during drilling operations, leading to a larger fracture width and resulting in a higher amount of fluid lost into the formation. When encountering a fracture while drilling the well, the fluid loss usually begins with partial loss, and encountering more extensive fractures will result in severe loss and even complete loss. In this research, the effect of fracture width on plugging efficiency was investigated using different fracture width sizes: 1.5, 2.0, and 3.0 mm. The formulation used in this investigation consisted of LT-RPPG with a ratio of 1:10. The waiting time for this test was 24 hours, and the fracture widths were 3.0, 2.0, and 1.5 mm.

Figure 10 shows the results of sealing pressure when different fracture widths were used. The highest sealing pressure was observed when the smallest fracture width was used. This indicates that this formulation has more effect on smaller fractures. However, using

lower swelling ratios will form a gel that can withstand higher pressure even in high fracture widths. Figure 11 shows the Frr results for sample 2 at different fracture widths using different flow rates.

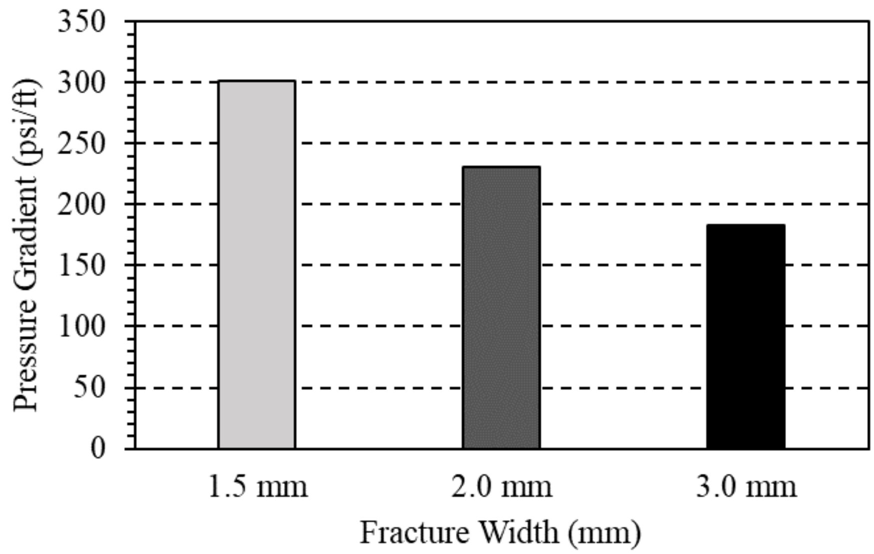

Figure 10. Sealing pressure at different fracture widths

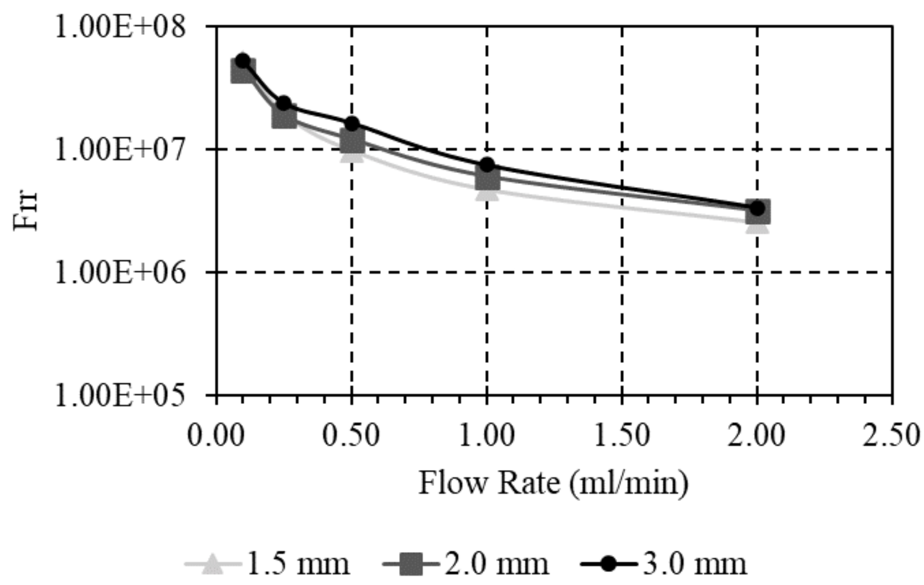

Figure 11. The Frr results for different fracture widths at different flow rates.

5.2.2. Swelling Ratio Effect. One of the critical factors that affects the strength of RPPG is the swelling ratio. Decreasing the swelling ratio will increase the strength. In this study, different swelling ratios were investigated, including 16, 10, 8, and 5. The waiting time for these tests was 24 hours, and the fracture width was 2.0 mm. Figure 12 presents the sealing pressure for each swelling ratio, showing that the sealing pressure increased with the decrease of the swelling ratio. This increase in sealing pressure is due to the increased amount of the LT-RPPG, resulting in a more robust material; hence, it will require higher pressure to break it. Figure 13 shows the Frr results for each flowing ratio at different flowing rates. Overall, the gel can reduce the permeability of fracture up to 10^8 times.

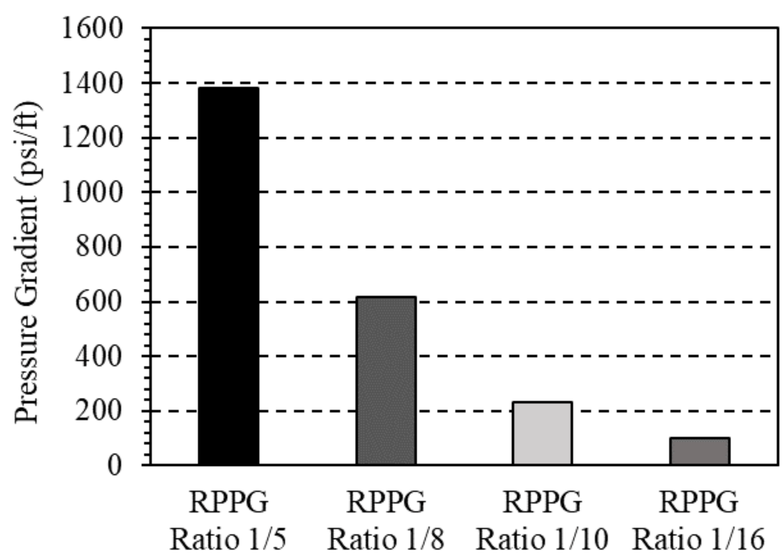

Figure 12. Sealing pressure at different swelling ratios

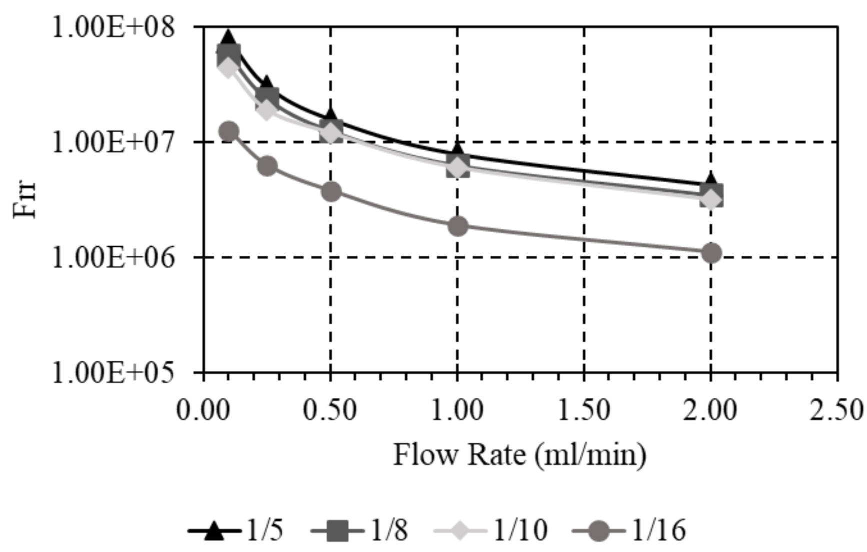

Figure 13. The Frr results for different swelling ratios at different flow rates

5.2.3. Waiting Time Effect. After injecting the sample into the fractured cemented core, it was left for a period of time for gel recrosslinking, which is referred to as the waiting time. In this research, five different waiting times were investigated, including 1, 6, 12, 24, and 48 hours. In these experiments, the swelling ratio of 16 was used along with a fracture width of 3.00 mm. Figure 14 shows the sealing pressure for different waiting times. The results showed that increasing the waiting time resulted in higher sealing pressures. This increase of sealing pressure is due to the material having more time to fully recrosslink, allowing for the sample to gain more strength, which reflects on its plugging ability. However, when the waiting time increased to 48 hours, the sealing pressure increased slightly, indicating that it is less effective in a waiting time beyond 24 hours. Figure 15 shows the Frr results for each waiting time at different flow rates.

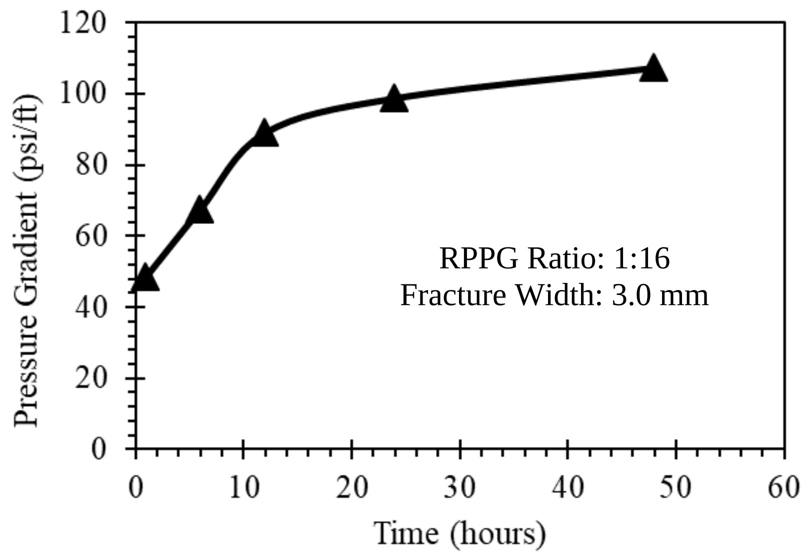

Figure 14. Sealing pressure for different waiting times

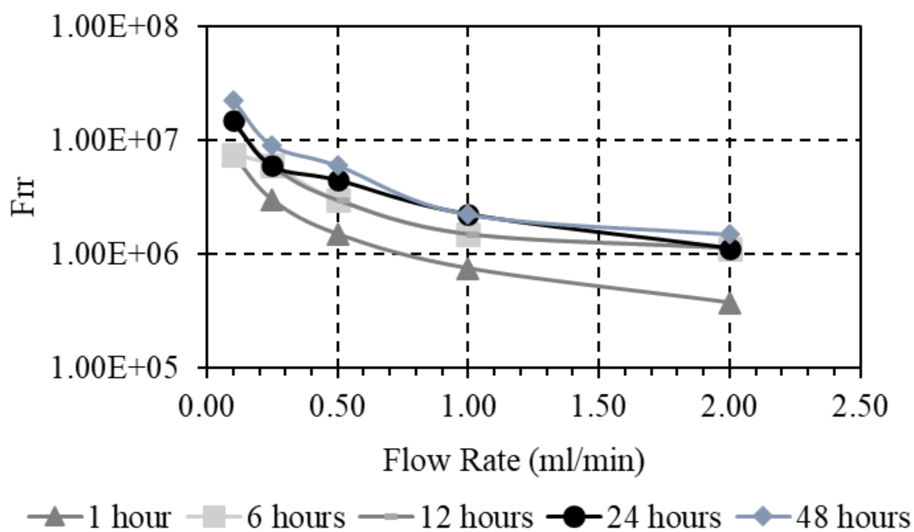

Figure 15. The Frr results for different waiting time at different flow rates

6. DISCUSSION

A series of experiments was conducted to evaluate the performance of the LT-RPPG as a lost circulation material. Table 2 summarizes the results of the sealing pressure for different swelling ratios LT-RPPG when different fracture widths were used. The highest sealing pressure obtained was 1381.51 psi/ft when the swelling ratio five was used for the 2 mm fracture. The lowest pressure was 48.37 psi/ft when using the swelling ratio 16 with the very shortest waiting time (1 hour) and fracture width of 3.0 mm. In fact, the product used in this study is a commercial product and its recrosslinking time is set. In real applications, the RPPG recrosslinking time can be adjusted when the product is synthesized. A RPPG product with a short recrosslinkable time should be easily made based on field requirements. Therefore, the waiting time should not be the major factor to be considered in the evaluation. This product has been successfully applied in the West Sak field on the North Slope of Alaska for enhancing the waterflood profile modification by treating void space conduits (Targac et al., 2020).

Moreover, we can see that the RPPG treatment can significantly reduce fracture permeability more than 10^6 times. As mentioned before, the RPPG can remain in the fracture even after the breakthrough, which reduces the permeability in the fractures. When the swelling ratio 5 was used, the permeability could be reduced to as low as 21.2 md when the fracture width of 2.0 mm was used, compared to 3.37×10^5 Darcy before treatment, and 33.66 md for 3.0 mm fracture width, compared to 7.59×10^5 Darcy before treatment.

Table 2. Sealing pressure results at different fracture widths

Group	LT-RPPG Ratio	Fracture Width mm	Waiting Time (hours)	Sealing Pressure Psi/ft	Permeability (Darcy)	
					Before Treatment	After Treatment
#1	1:16	1.5	24	243.10	$1.90{\times}10^5$	0.0347
#1	1:16	2	24	100.75	$3.37{\times}10^5$	0.0885
#1	1:16	3	24	98.82	7.59×105	0.1683
#1	1:16	3	1	48.37	7.59×105	0.5049
#1	1:16	3	6	67.50	7.59×105	0.2524
#1	1:16	3	12	89.02	7.59×105	0.2524
#1	1:16	3	48	107.16	7.59×105	0.1262
#2	1:10	1.5	24	301.20	1.90×105	0.0192
#2	1:10	2	24	230.97	$3.37{\times}10^5$	0.0279
#2	1:10	3	24	183.04	$7.59{\times}10^5$	0.0459
#3	1:8	2	24	615.96	$3.37{\times}10^5$	0.0265
#3	1:8	3	24	374.43	$7.59{\times}10^5$	0.0421
#4	1:5	2	24	1381.51	$3.37{\times}10^5$	0.0212
#4	1:5	3	24	1074.91	$7.59{\times}10^5$	0.0337

Ravi et al. (2006) found that the sealing pressure of the rubbery polymer with graphitic carbon and laminate particles treatment could reach up to 1390 psi when a fracture width of 1 to 3 mm was used. Our material can reach a pressure of 1381.51 psi/ft when a 1:5 ratio is used with 2 mm of fracture width without any additives. Also, when Ravi et al. (2006) used a clay-swelling chemical treatment, with and without additives, they obtained a sealing pressure ranging from 537 to 1298 psi when 1-3 mm fractures were used and found 462 psi when 2-3 mm fractures were used. Jia et al. (2021) investigated a novel high-density bromide-based nano-composite gel (HDGEL). They used a fracture width of 2.1 mm, and the highest sealing pressure they obtained was 56.56 psi (344.67 psi/ft) compared to our result, which was 1381.51 psi/ft, and 615.96 psi/ft when the swelled ratio of 5 and 8 were used, respectively. The LT-RPPG showed its capability to be an excellent candidate to mitigate losses in the absence of drilling fluid. However, one concern is whether drilling

fluids could damage the performance. We are conducting a series of new experiments to address the concerns and our preliminary results have shown drilling fluids can significantly enhance the performance. The comprehensive research results regarding the combination will be reported in a separate paper for better explaining all results.

7. CONCLUSION

A comprehensive study was performed to investigate the capability of LT-RPPG to control fluid loss. Three factors, including the gel particle swelling ratio, fracture width, and waiting time, were studied to evaluate their impacts on the gel strength, sealing pressure, and plugging efficiency. Based on this study, the LT-RPPG is an excellent candidate to work as a fluid loss control material during drilling operations. A summary of the findings deduced from this research is as follows:

- The LT-RPPG which can recrosslink to form a strong bulk gel after being placed in fracture showed promising results to control fluid loss during drilling.
- The fracture width is inversely proportional to sealing pressure, as the sealing pressure reached 1074 psi/ft when the fracture width was 3.0 mm and 1381.5 psi/ft when the fracture width was 2.00 mm when a swelling ratio of 5 was used.
- The LT-RPPG swelling ratio is an important factor. Reducing swelling ratios will result in more robust material, which leads to higher sealing pressure as the sealing pressure reached 1381 psi/ft when a swelling ratio of 5 was used for the fracture with a width of 2.0 mm.

- Waiting time plays an essential role in the sealing efficiency, as the LT-RPPG requires sufficient time to fully recrosslink to gain higher strength, which reflects on its ability to withstand higher pressure. However, the recrosslinking time is adjustable when a specific case is available.
- For all of the experiments, the fracture permeabilities were reduced more than 10^6 times after the treatment, indicating that the LT-RPPG has the potential of enhancing the plugging efficiency.

NOMENCLATURE

RPPG	Re-Assembling Preformed Particle Gel
KCl	Potassium Chloride
SEM	Scanning Electron Microscope
Frr	Residual Resistance Factor
HPAM	Hydrolyzed Polyacrylamide Polymer
PEI	Polyethleneimine
GP-A	Hydrophobic Association Supramolecular Hydrogel
ECS-1	Engineered Composite Solutions
DPP	Deformable Plugging Polymer
AMPS	2-Acrylamide-2-Methylpropionic Sulfonic Acid
SPAM	High-Temperature Resistance Acrylamide Based Polymer
SR	Swelling ratio

REFERENCES

Al Maskary, S., Halim, A. A., & Al Menhali, S. (2014, November). Curing losses while drilling & cementing. In Abu Dhabi International Petroleum Exhibition and Conference. OnePetro. doi: https://doi.org/10.2118/171910-MS

Almagro, S. P. B., Frates, C., Garand, J., & Meyer, A. (2014). Sealing fractures: Advances in lost circulation control treatments. Oilfield Review, 26(3), 4-13.

Alsaba, M., Nygaard, R., Hareland, G., & Contreras, O. (2014, April). Review of lost circulation materials and treatments with an updated classification. In AADE National Technical Conference and Exhibition, Houston, TX (pp. 1-9).

Bao, D., Qiu, Z., Zhao, X., Zhong, H., Chen, J., & Liu, J. (2019). Experimental investigation of sealing ability of lost circulation materials using the test apparatus with long fracture slot. Journal of Petroleum Science and Engineering, 183, 106396.

Canson, B. E. (1985, March). Lost circulation treatments for naturally fractured, vugular, or cavernous formations. In SPE/IADC drilling conference. OnePetro. doi: https://doi.org/10.2118/13440-MS

Cook, J., Growcock, F., Guo, Q., Hodder, M., & van Oort, E. (2011). Stabilizing the wellbore to prevent lost circulation. Oilfield Review, 23(4), 26-35.

Gamage, P., Deville, J. P., & Sherman, J. (2014). Solids-free fluid-loss pill for high-temperature reservoirs. SPE Drilling & Completion, 29(01), 125-130.

Gang, W., Honghai, F., Jie, F., Wanjun, L., Yu, Y., Xiangji, K., ... & Haijun, Y. (2020). Performance and application of high-strength water-swellable material for reducing lost circulation under high temperature. Journal of Petroleum Science and Engineering, 189, 106957.

Hashmat, M. D., Sultan, A. S., Rahman, S., & Hussain, S. M. (2016, April). Crosslinked polymeric gels as loss circulation materials: An experimental study. In SPE Kingdom of Saudi Arabia Annual Technical Symposium and Exhibition. OnePetro. doi: https://doi.org/10.2118/182740-MS

Hettama, M., Horsrud, P., Taugbol, K., Friedheim, J., Huynh, H., Sanders, M. W., & Young, S. (2007, March). Development of an innovative high-pressure testing device for the evaluation of drilling fluid systems and drilling fluid additives within fractured permeable zones. In Offshore Mediterranean Conference and Exhibition. OnePetro.

Howard, G. C., & Scott, P. P. (1951). An analysis and the control of lost circulation. Journal of Petroleum Technology, 3(06), 171-182.

Jeennakorn, M. (2017). The effect of testing conditions on lost circulation materials' performance in simulated fractures. Missouri University of Science and Technology.

Jia, H., Chen, H., & Guo, S. (2017). Fluid loss control mechanism of using polymer gel pill based on multi-crosslinking during overbalanced well workover and completion. Fuel, 210, 207-216.

Jia, H., Kang, Z., Zhu, J., Ren, L., Cai, M., Wang, T., & Li, Z. (2021). High density bromide-based nanocomposite gel for temporary plugging in fractured reservoirs with multi-pressure systems. Journal of Petroleum Science and Engineering, 205, 108778.

Jiang, G., Deng, Z., He, Y., Li, Z., & Ni, X. (2019). Cross-linked polyacrylamide gel as loss circulation materials for combating lost circulation in high temperature well drilling operation. Journal of Petroleum Science and Engineering, 181, 106250.

Kefi, S., Lee, J. C., Shindgikar, N. D., Brunet-Cambus, C., Vidick, B., & Diaz, N. I. (2010, November). Optimizing in four steps composite lost-circulation pills without knowing loss zone width. In IADC/SPE Asia Pacific Drilling Technology Conference and Exhibition. OnePetro. doi: https://doi.org/10.2118/133735-MS

Kumar, A., & Savari, S. (2011, April). Lost circulation control and wellbore strengthening: looking beyond particle size distribution. In AADE national technical conference and exhibition, Houston, Texas, USA (pp. 12-14).

Kumar, A., Savari, S., Whitfill, D. L., & Jamison, D. E. (2010). Wellbore Strengthening: The Less-Studied Properties of Lost-Circulation Materials. Paper SPE 133484 presented at SPE Annual Technical Conference and Exhibition. Florence, Italy, 19-22 September.

Li, J., Qiu, Z., Zhao, X., Zhong, H., Yang, Y., & Huang, W. (2020). Effect of different parameters on sealing performance of lost circulation process with dynamic fracture aperture testing apparatus. Journal of Petroleum Science and Engineering, 193, 107357.

Magzoub, M. I., Shamlooh, M., Salehi, S., Hussein, I., & Nasser, M. S. (2021). Gelation kinetics of PAM/PEI based drilling mud for lost circulation applications. Journal of Petroleum Science and Engineering, 200, 108383.

Mansour, A., Taleghani, A. D., Salehi, S., Li, G., & Ezeakacha, C. (2019). Smart lost circulation materials for productive zones. Journal of Petroleum Exploration and Production Technology, 9(1), 281-296.

Moore, P. L. (1986). Drilling practices manual.

Nayberg, T. M., & Petty, B. R. (1986, April). Laboratory study of lost circulation materials for use in oil-base drilling muds. In SPE Deep Drilling and Production Symposium. OnePetro.

Pu, J., Bai, B., & Schuman, T. (2021). U.S. Patent No. 11,162,016. Washington, DC: U.S. Patent and Trademark Office.

Pu, J., Bai, B., Alhuraishawy, A., Schuman, T., Chen, Y., & Sun, X. (2019). A recrosslinkable preformed particle gel for conformance control in heterogeneous reservoirs containing linear-flow features. SPE Journal, 24(04), 1714-1725. doi: https://doi.org/10.2118/191697-PA

Ravi, K., Savery, M. R., Reddy, B. R., & Whitfill, D. L. (2006, October). Cementing technology for low fracture gradient and controlling loss circulation. In SPE/IADC Indian Drilling Technology Conference and Exhibition. OnePetro. doi: https://doi.org/10.2118/102074-MS

Sanders MW, Scorsone JT and Friedheim JE, (2010): High-fluid-loss, high-strength lostcirculation treatments. SPE Deepwater Drilling and Completions Conf., Galveston, Texas, USA;5-6 October, SPE Paper#135472

Sanders, M. W., Young, S., & Friedheim, J. (2008, April). Development and testing of novel additives for improved wellbore stability and reduced losses. In AADE fluids conference and exhibition, Houston, USA (pp. 8-9).

Savari, S., & Whitfill, D. L. (2019, November). Managing lost circulation in highly fractured, vugular formations: engineering the LCM design and application. In Abu Dhabi International Petroleum Exhibition & Conference. OnePetro. doi: https://doi.org/10.2118/197186-MS

Song, T., Feng, Q., Schuman, T., Cao, J., & Bai, B. (2021). A Novel Branched Polymer Gel System with Delayed Gelation Property for Conformance Control. SPE Journal, 1-11. doi: https://doi.org/10.2118/206742-PA

Targac, G., Gallo, C., Smith, D., Huang, C. K., Autry, S., Peirce, J., & Baohong, L. (2020, October). Case History of Conformance Solutions for West Sak Wormhole/Void Space Conduit with a New Reassembling Pre-formed Particle Gel RPPG. In SPE Annual Technical Conference and Exhibition. OnePetro. doi: https://doi.org/10.2118/201302-MS

Van Oort, E., Friedheim, J., Pierce, T., & Lee, J. (2011). Avoiding losses in depleted and weak zones by constantly strengthening wellbores. SPE Drilling & Completion, 26(04), 519-530.

Wang, B., Sun, J., Lv, K., Shen, F., & Bai, Y. (2021). Effects of a crosslinking agent on a supramolecular gel to control lost circulation. New Journal of Chemistry, 45(16), 7089-7095.

Wang, L., Long, Y., & Bai, B. (2018). Low temperature applicable polyelectrolyte gelator to covalently bridged partially hydrolyzed poly (acrylamide) in situ gel for fossil energy recovery. Chemical Engineering Journal, 334, 2558-2567.

White, R. J. (1956, January). Lost-circulation Materials and their Evaluation. In Drilling and Production Practice. OnePetro.

Whitfill, D., & Miller, M. (2008, April). Developing and testing lost circulation materials. In AADE fluids conference and exhibition, Houston, Texas, USA (pp. 8-9).

Xu, C., Zhang, J., Kang, Y., You, L., Yan, X., Cui, K., & Lin, C. (2019, March). Investigation on the transport and capture behaviours of lost circulation material in fracture with rough surface. In International Petroleum Technology Conference. OnePetro.

PAPER

II. IMPACT OF DIFFERENT ADDITIVES ON LOW-TEMPERATURE RECROSSLINKABLE PREFORMED PARTICLE GEL TO MITIGATE FLUID LOSSES IN FRACTURED FORMATIONS

ABSTRACT

During drilling through highly permeable or fractured formations, the entire drilling fluid or most of it might be lost into formations; this phenomenon is called lost circulation. Lost circulation can cause serious issues during drilling, such as dangerous well control difficulties, and, in some cases, complete loss of the well. In this paper, a Low-Temperature Recrosslinkable Preformed Particle Gel (LT-RPPG) will be evaluated in the presence of various additives to control drilling fluid loss in fractured formations. Different factors were investigated, including mica concentrations, mica particle size, walnut shell concentrations, and bentonite. Core flooding experiments were conducted to evaluate its plugging efficiency utilizing fractured cement cores. The results show that mixing the LT-RPPG with drilling fluids provides a higher-strength material which is reflected in a higher sealing pressure than when using brine only. Also, introducing walnut shell to the mixture results in more robust materials and significantly improving the sealing pressure for 3.0 mm fracture width up to 1104.97 psi/ft when 0.75% walnut shells were used. The fracture permeability can reduce more than 10^8 times even if the gel is broken down by subsequent circulation of drilling fluid.

1. INTRODUCTION

Lost circulation is a significant and prevalent issue when drilling through natural fractured, highly permeable, or cavernous formations. Usually, the drilling operations induce these fractures and result in the full loss of returns (Moore, 1986). Drilling fluid losses are classified based on loss volume to seepage loss lower than 10 bbl/hr, partial loss between 10 to 20 bbl/hr, severe loss when losses exceed 200 bbl/hr, and full loss of return (Nayberg & Petty, 1986). Lost circulation can lead to a drastic cost increase as it significantly increases the nonproductive time (NPT), which increases the operational cost (Kumar et al., 2010). The annual cost problems regarding lost circulation are around one to four billion dollars (Cook et al., 2011; Sanders et al., 2010; Al Maskary et al., 2014). Moreover, lost circulation can result in well control issues, leading to a potential blowout. To overcome these issues, lost circulation materials (LCM) have been used. Firstly, these materials were classified into fibrous, flaky, and granular or a blend of the three based on their appearance or physical properties (Howard and Scott, 1951; Ramasamy, et al., 2018). Lately, a new classification has been introduced based on their physical and chemical properties and their applications. They have now been classified into seven categories including granular, flaky, fibrous, a mixture of LCM’s, acid-soluble/water-soluble, high-fluid loss LCMs squeeze, swellable/hydratable LCM combinations, and nanoparticles (Alsaba et al., 2014).

There are different methods used to evaluate the performance of LCM treatments, including filtration tests utilizing slotted disks and sealing efficiency tests utilizing fractured cores. The most common apparatus for the filtration tests is a particle plugging

apparatus (PPA) with slotted disks to represent the fractured formation (Whitfill and Miller, 2008).

Different materials have been tested in order to mitigate loss circulation issues. Howard and Scott (1951) studied the sealing ability of various LCMs at room temperature, and they found that granular materials are more suitable for plugging large fractures. Hinkebein et al. (1983) studied different conventional LCMs, and they found that as the LCM concentrations increase, the more likely they were to obtain successful sealing. Hashmat et al. (2016) used a transparent tube filled with glass beads to investigate the HPAM/PEI performance to seal highly permeable formations, 306 and 2965 Darcy. Their results showed that the HPAM/PEI gel succeeded in preventing mud loss at room temperature for the permeability of 306 Darcy, however, it could not prevent the mud loss for permeability of 2965 Darcy. Bao et al. (2019) conducted experimental studies to investigate the capability of different LCM materials to plug fractures. Their results indicated that increasing the concentrations of LCM will result in higher sealing pressure. The impact factors of particle size distribution and particle concentrations on sealing efficiency were investigated by Xu et al. (2019) and Li et al. (2020). Xie et al. (2021) studied cross-linked modified polyacrylamide gel (HMP gel) for lost circulation treatments. They used a customized design to test the plugging efficiency utilizing fracture slots. Their results indicated that HMP gel could withstand a pressure of 700 psi and 900 psi when fracture widths of 2 and 1 mm were used. Lashkari et al. (2021) studied shape memory polyurethane for LCM applications. They found the shape memory polyurethane could seal the wide fracture once activated by formation reservoirs as it withstood the pressure of 1450 psi at 80 °C. Cui et al. (2021) evaluated a shape memory polymer (SMP)

to prevent losses by using a high-temperature, high-pressure water loss apparatus. They increased the pressure by 50 psi every five minutes, and they found that after adding 2% of SMP, it could withstand pressure up to 300.2 psi at room temperature. Qiu et al. (2022) studied an oil-based polymer gel to control fluid loss at high temperature conditions. They found that this polymer gel could withstand up to 464 psi when a fracture width of 3.0 mm was used at 160 °C.

For the last decades, several studies were conducted to evaluate the LCMs, yet they are still found a lack of capability in preventing or mitigating severe loss (Du et al., 2017). Hence, introducing a new material is important to terminate or reduce the impact on drilling operations. In our previous study (Ahdaya et al., 2022), a new material, LT-RPPG, was introduced and evaluated to be used as lost circulation material. Based on the obtained results, we found that the LT-RPPG is a promising material to cure severe loss. Thus, a new evaluation was conducted in this research to investigate its capability to mitigate losses in the presence of different additives such as drilling fluid, mica, and walnut shell.

2. EXPERIMENTAL MATERIALS DESCRIPTION

Drilling Fluid. The drilling fluid used in this research is a water-based mud with 7% bentonite. The drilling fluid was mixed using a Hamilton Beach mixer for at least 10 minutes and then was left for at least 18 hours for bentonite prehydration to occur to ensure that the bentonite was fully swelled (Ahdaya and Imqam, 2019).

Brine**.** 2% potassium chloride (KCl) was used as a base fluid for all samples.

Low temperature re-assembling preformed particle gel. LT-RPPG is a dry, white granular particle consisting of a crosslinked poly (acrylamide-co-acrylic acid) copolymer

with a pre-embedded second crosslinker (Crosslinker II) (PU, et al., 2019). The LT-RPPG can be stable at temperature up to 80 °C. The preparation of the LT-RPPG was formulated by adding the desired amount of the LT-RPPG particle to the 2% KCl solution and mixed. After swelling the gel slurry is weakly acidic.

LCM mica. LCM mica is a type of flaky material that is used to reduce fluid loss during drilling operations. The chemical compositions of the LCM mica used in this research are shown in Table 1. There were different types of LCM mica used based on particle size distribution, including fine, medium, and coarse mica. Table 2 shows how the mica particle size ranges for each type. LCM mica was provided by the Pacer Minerals company.

Table 1. The chemical compositions of mica

Component	**Wt. %**
SiO_2	64.7
Al_2O_3	16.3
Fe_2O_3	2.6
K_2O	4.6
Na_2O	1.4
CaO	0.3
MgO	5.5
L.O.I	2.0

Table 2. Mica particle size distribution

Particle size (Microns)	**Fine mica (%)**	**Medium mica (%)**	**Coarse mica (%)**
< 149	28.70	16.30	4.60
149 to 250	21.90	21.00	14.50
250 to 850	41.80	52.10	52.30
850 to 1400	7.60	10.60	15.40
1400 to 3350	0.0	0.0	13.20
> 3350	0.0	0.0	0.0

Walnut shell. Walnut shell is a type of granular material that is used to control fluid loss during drilling. A fine walnut shell with particle ranges < 590 microns was used in this research.

Cement cores. The fractured cement cores were prepared by mixing tap water with cement powder and were then poured into the cylinder molds. A steel bar was utilized to create the fracture inside the core during the hydration process. A fracture of 3.0 mm width was used in this study.

HAAKE rheometer**.** A HAAKE rheometer was used in this study to measure the elastic modulus (G') and viscous modulus (G'') of each sample to better understand the effect of each parameter on the material's strength.

Scanning electron microscopy (SEM). The microstructure of LT-RPPG was characterized with HELIOS NANO LAB 600. The swelled LT-RPPG samples were freeze-dried and coated before testing.

3. EXPERIMENTAL SETUP DESCRIPTION

Figure 1 shows the setup design that was used to perform the experiments. The design consists of a core holder to hold the fractured cement core, an accumulator which contains the sample and is used to inject the sample into the fractured cement core, a pressure transducer that connects to the computer to record pressure changes during the experiment, and a syringe pump to apply the flow rates. All the experiment were conducted at room temperature.

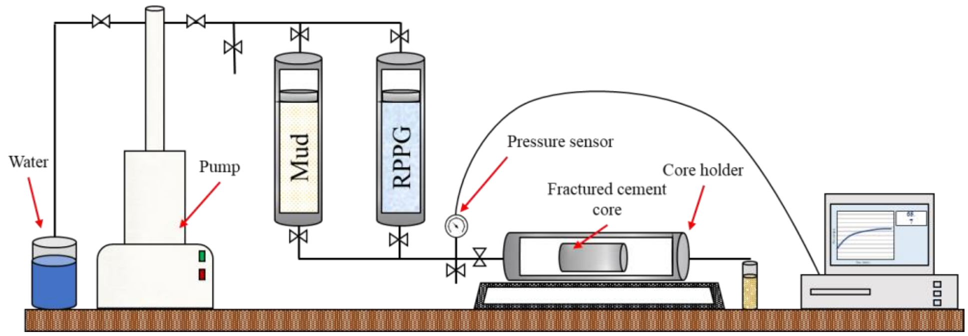

Figure 1. Core flooding setup design

4. EXPERIMENTAL PROCEDURE DESCRIPTION

Sealing pressure (also called breakthrough pressure) and Residual Resistance Factor (*Frr*) were evaluated using core flooding tests. The sealing pressure is known as the highest pressure that a material can withstand before it cracks or collapses. The exact procedure of these experiments consists of the following:

1) The fractured cement core was placed in the core holder, and confining pressure was applied.
2) The drilling fluid was injected into the fractured core to wet the fracture surfaces to simulate the actual events in the actual wells.
3) The tested sample was injected into the fracture using a constant flow rate of 1 mL/min, and the injection was stopped when the pressure was stabilized.
4) Then it was left for 24 hours for recrosslinking processes.
5) At that point, the drilling fluid was injected at a flow rate of 1 mL/min to measure the sealing pressure of the material.

After the sealing pressure experiment was concluded, *Frr* was measured. The following procedure was conducted to measure *Frr*:

1) The accumulator was filled with the brine and injected into the fractured core.
2) The injection flow rates used were 0.1, 0.25, 0.50, 1.00, and 2.00 mL/min.
3) The permeability was calculated at each flow rate using the Darcy equation:

$$k = \frac{q\,\mu\,L}{A\,\Delta P} \quad (1)$$

Where, K is permeability, Darcy, Q is flow rate, mL/sec, μ is viscosity, cp, L is core length, cm, A is cross-section area, cm^2, and ΔP is differential pressure, atm.

Then, the Frr was calculated using the following equation:

$$F_{rr} = \frac{K_{before}}{K_{after}} \quad (2)$$

Where, K_{before} is the permeability of the fracture before the treatment, and K_{after} is the permeability of the fracture after the treatment.

5. RESULTS AND DISCUSSION

5.1. INTERACTIONS BETWEEN LT-RPPG AND DRILLING FLUID

Different drilling fluids with different bentonite concentrations were investigated in this research to better understand the effect of drilling fluid on the LT-RPPG. Figure 2 shows a comparison of the G' and G'' for samples in the presence and absence of drilling fluid. The base formulation in these mixtures is LT-RPPG with a swelling ratio of 16 and 2% KCL. Results showed that adding the drilling fluid increased the strength of the sample.

The G' and G'' were 235.45 and 88.56 Pa, respectively, when no drilling fluid was present. However, they increased to 321.52 and 96.22 Pa when a drilling fluid with 7% bentonite was used. This indicates that mixing the LT-RPPG with drilling fluid provides a stronger material than does the use of brine. Figure 3 shows the SEM image for a sample that was prepared with drilling fluid and LT-RPPG with a swelling ratio of 10 after being fully recrosslinked. The 3D network structures showed strong bonding and highly compacted pore size.

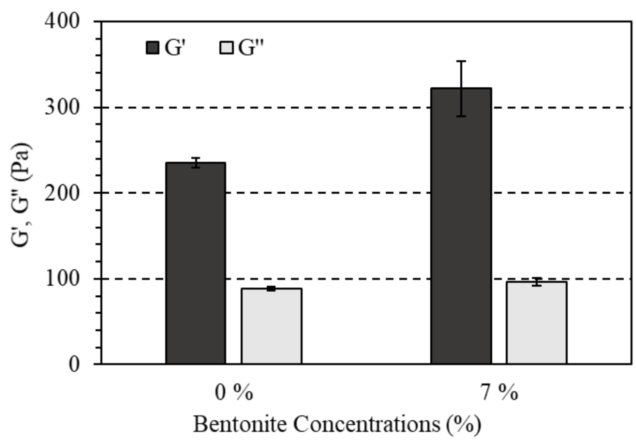

Figure 2. The results of G' and G" at different concentrations of bentonite.

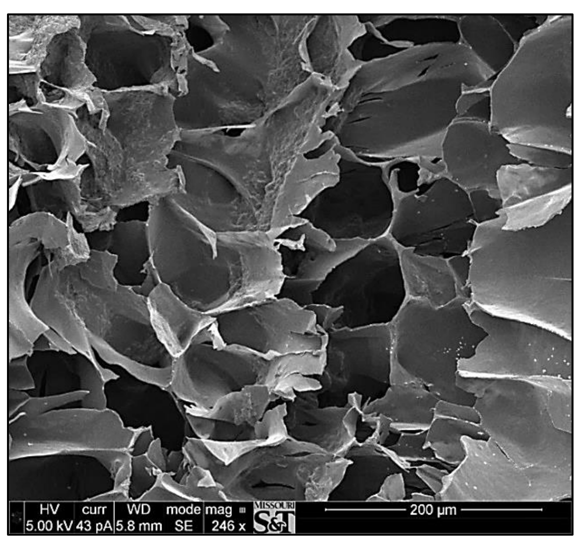

Figure 3. SEM image of LT-RPPG swelled in drilling fluid

5.2. INTERACTION BETWEEN LT-RPPG, DRILLING FLUID, AND MICA

In this section, the effect of mixing the drilling fluid and mica with LT-RPPG is described. Table 3 shows some of the mixing designs used to investigate the effect of mica on LT-RPPG. Different concentration of micas were added to the samples to evaluate their performance. In addition, different mica particle sizes were investigated to understand their impact on the strength of the mixture. The samples were prepared by adding the LT-RPPG and mica to the drilling fluid.

Table 3. LT-RPPG with mica and drilling fluid mixing design

#	Drilling fluid		LT-RPPG Ratio	Mica Concentrations (%)	Mica Type
	Bentonite (%)	Water			
Sample 1	7	2 % KCl	1/16	1	Coarse
Sample 2	7	2 % KCl	1/16	1	Medium
Sample 3	7	2 % KCl	1/16	2	Coarse
Sample 4	7	2 % KCl	1/10	1	Coarse
Sample 5	7	2 % KCl	1/16	1	Fine
Sample 6	7	2 % KCl	1/16	1	Coarse
Sample 7	0	2 % KCl	1/16	1	Coarse
Sample 8	7	2 % KCl	1/16	0	-

Figure 4 shows sample 1. The sample was in the liquid phase, and the LT-RPPG and mica particles were suspended after the mixing was completed. The mixture started to recrosslink after about 20 minutes. The G’ and G’’ were 1031.59 and 319.8 Pa, respectively. Figure 5 shows sample 2. The sample was in the liquid phase after the mixing was completed. After 80 minutes, the mixture started to recrosslink. The G’ was 971.74 Pa, and the G’’ was 298.93 Pa. Figure 6 shows the G’ and G’’ when different mica types were used. Mica particle size has a significant impact on the strength of the material, as the

results showed that the G’ was 442 Pa when fine mica was used compared to 1031 Pa when coarse mica was used. This indicates that the mixture gained higher strength when larger particles were introduced. Since the mica includes alumina, it reacts with the weakly acidic LT-RPPG and generates aluminum cation, which is also a metal cross-linker, that further enhances the cross-linking and leads to higher strength (Borling et al., 1994).

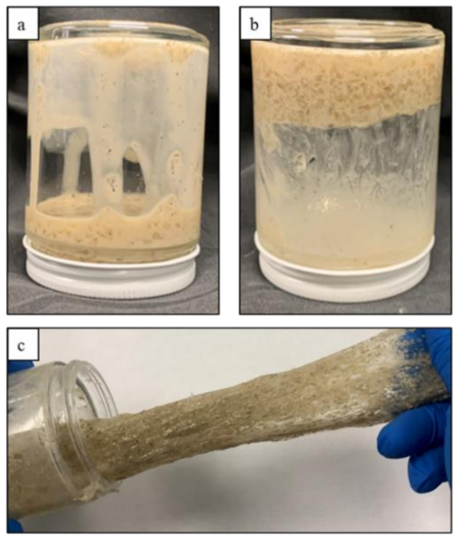

Figure 4. Sample 1 (a) immediately after mixing. (b) after 24 hours. (c) after 48 hours

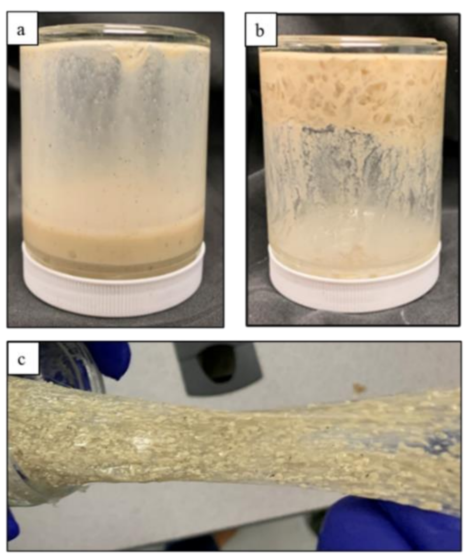

Figure 5. Sample 2 (a) immediately after mixing. (b) after 24 hours. (c) after 48 hours

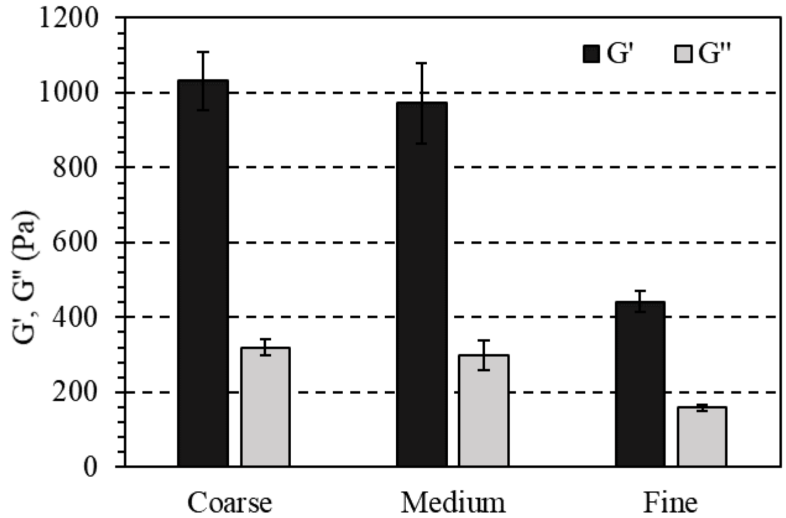

Figure 6. G’ and G” for different mica type.

Figure 7 shows the G’ and G” results when different mica concentrations were used. The results showed that when 1.00% of mica was added to the mixture, the G’ increased significantly as it was 321 Pa when no mica was present, compared to 1031 Pa when 1% of coarse mica was introduced. However, when the amount of mica was increased to 2%, the G’ reduced slightly to 937 Pa.

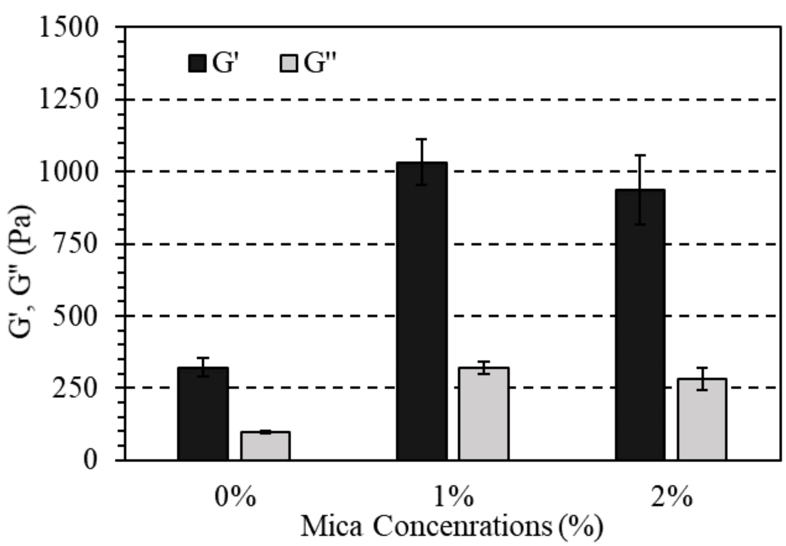

Figure 7. G’ and G” for different mica concentrations.

5.3. INTERACTION BETWEEN LT-RPPG AND WALNUT SHELL.

This section will describe the effect of mixing the LT-RPPG and walnut shells. Different walnut shells concentrations were added to the samples to investigate their impacts on the LT-RPPG strength. The samples were prepared by adding the LT-RPPG and walnut shell particles to the brine solution. Figure 8 shows a sample consisting of LT-RPPG with a ratio of 10 and 1% walnut shell. The LT-RPPG particles absorbed all the brine solution after about 30 minutes of mixing and started recrosslinking five minutes later. The G' was around 820.95 Pa, and the G" was 257 Pa. The walnut shell particles suspended in the mixture, as illustrated in the picture, could aggregate in the fracture during the injection and form a mat-like plug with the LT-RPPG to plug the fracture permanently. Figure 9 shows the G' and G" in the presence and absence of walnut shells. Adding the walnut shell to the sample contributed to the increase of the strength slightly, as the G' increased from 706.55 Pa to 820.95 Pa when 1% of walnut shells was introduced. Figure 10 shows the SEM image for the sample after adding 1% of fine walnut shells. The image illustrates that the walnut shell did not affect the pore size structures, which are still strongly interconnected.

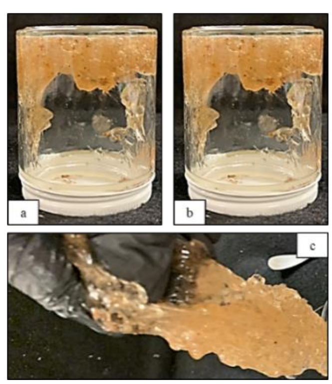

Figure 8. LT-RPPG mixture with walnut shell (a) immediately after mixing. (b) after 24 hours. (c) after 48 hours

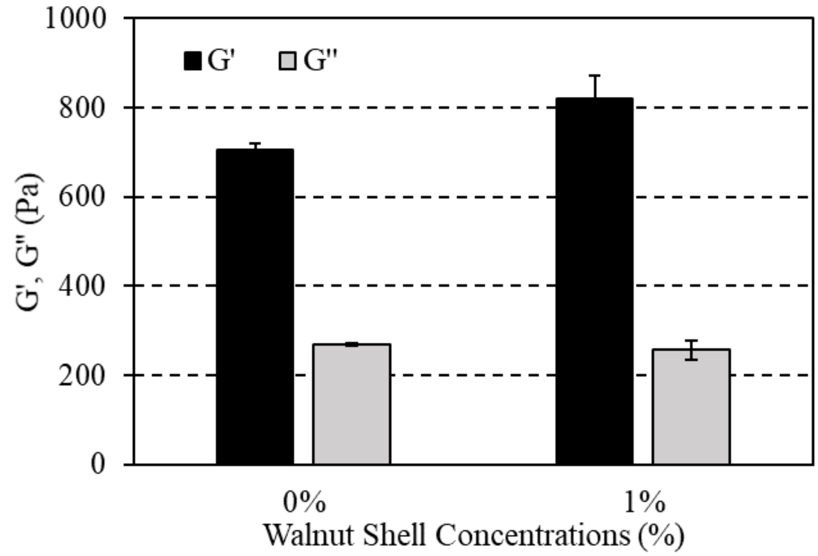

Figure 9. G' and G'' for different walnut shell concentrations.

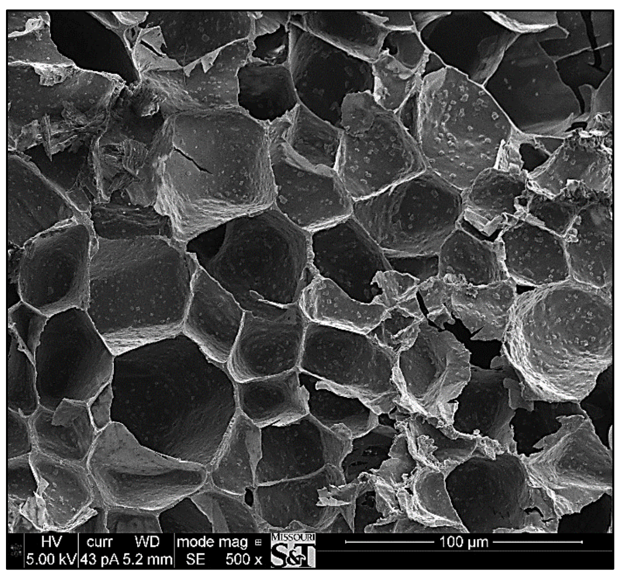

Figure 10. SEM image of LT-RPPG hybrid with walnut shell

5.4. CORE FLOODING EXPERIMENT RESULTS

To investigate the effect of different additives on the LT-RPPG plugging efficacy, various core flooding experiments were performed. These results will be presented in this section. An entire experiment will be described to show how the sealing pressure was

measured. The investigation was conducted using a sample of LT-RPPG with a swelling ratio of 16 prepared with drilling fluid (7% bentonite). The fracture width was 3.0 mm and at room temperature. Figure 11 shows the obtained results. The graph shows that the LT-RPPG injection pressure increases for the first 90 minutes and then stabilizes at about 80 psi/ft. After reaching stable pressure, the injection was stopped, and the core was left for recross-linking processes for 24 hours. Subsequently, the drilling fluid was injected to measure the sealing pressure. The sealing pressure for this experiment reached 94.65 psi/ft. The fractured cement core before and after the experiment is shown in Figure 12. The residual mixture inside the fracture is shown in Figure 13. The pictures illustrate the residual sample inside the fracture after the post-injection. This remaining gel reduces the fracture permeability significantly after the breakthrough, with more than 99.99% reduction, as it was around 7.6 E+08 md before treatment and became 5.52 md after treatment.

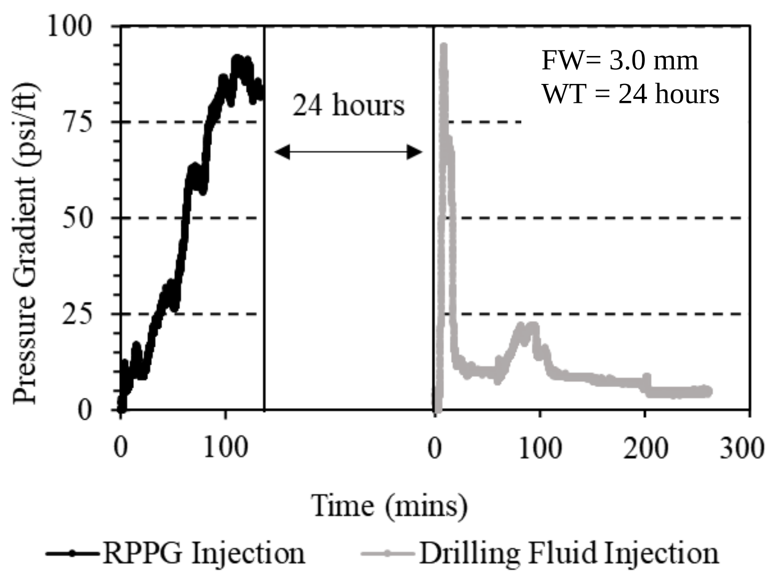

Figure 11. Core flooding results for LT-RPPG mixed with drilling fluid

Figure 12. Fractured cement core before and after the experiment

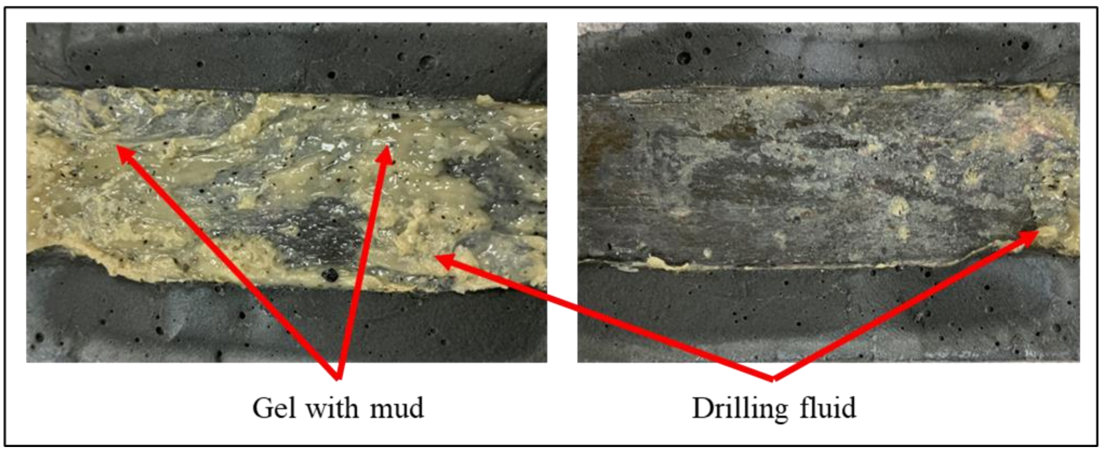

Figure 13. Inside image of the fracture for the fractured cement core after the experiment

5.4.1. Mica Concentration Effect. The concentration of mica was investigated in the study. Four different mica concentrations were used, including 0.50, 0.75, 1.00, and 2.00 wt.%. The base formulation used in this study consisted of drilling fluid, LT-RPPG with a swelling ratio of 16, and coarse mica. The waiting time was 24 hours, and the fracture width was 3.0 mm. Figure 14 shows the results of sealing pressure for different mica concentrations. The results indicate that mica concentrations are directly proportional

to sealing pressure. Therefore, an increase in the mica concentration raises the sealing pressure. For instance, the sealing pressure was 357.75 psi/ft when a concentration of 2.0 wt.% was used compared to 151.36 psi/ft when 0.5 wt.% was used. This increase in the sealing pressure is because adding more mica particles will increase the amount of the solids in the mixture, which will aggregate and form a more robust seal in the fracture (White, 1956). The permeability was reduced significantly after the treatment, as it was 7.59E+05 Darcy, and it decreased to 12.94, 6.01, 5.67, and 5.61 md when 0.50, 0.75, 1.00, and 2.00 wt% of mica were used, respectively. Figure 15 shows Frr results for different mica concentrations at different flow rates.

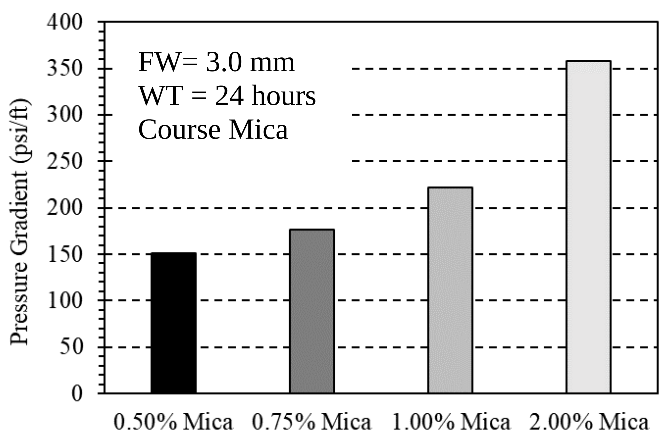

Figure 14. Sealing pressure at different mica concentrations

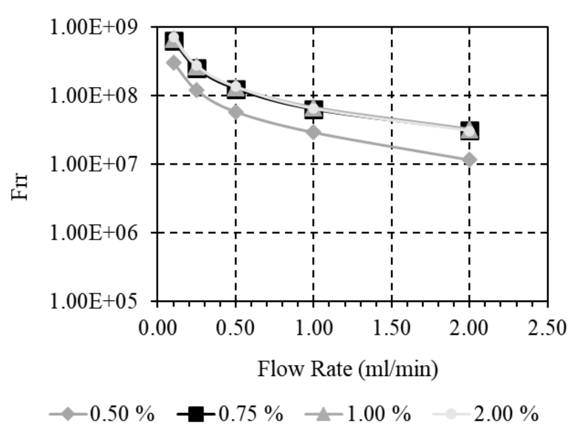

Figure 15. Frr results at different flow rates

5.4.2. Mica Particle Size Effect. The impact of mica particle size was studied. Three different mica particle sizes were used, including coarse mica, medium mica, and fine mica. An LT-RPPG with a swelling ratio of 16 was used as a base formulation for these experiments. The concentration of 1 wt.% was used when each mica was added to the mixture. One mixture of three types of mica was used to understand the effect of using three types of mica on the sealing efficiency. The waiting time was 24 hours, and the fracture width was 3.0 mm. Figure 16 shows the sealing pressure when different mica particle sizes were used. The results showed no significant difference in sealing pressure between coarse mica and medium mica, as it was 222.24 psi/ft when coarse mica was used compared to 227.66 psi/ft when medium mica was used. However, when fine mica was used, the sealing pressure decreased slightly compared to the sealing pressure when using medium and coarse mica. The sealing pressure was 182.21 psi/ft for fine mica compared to 222.24 and 227.66 psi/ft for coarse and medium, respectively. The permeability results indicate an enormous decrease after the treatment for all mica types. For medium and coarse mica, the permeability reduces to 5.67 and 5.20 md, respectively, which demonstrates almost no difference; however, using fine mica particles resulted in higher permeability.

5.4.3. Walnut Shell Concentrations Effect. In this study, three different walnut shell concentrations were used, including 0.50, 0.75, and 1.00%. The base formulation used for these experiments includes an LT-RPPG with a swelling ratio of 10 and walnut shell with fine particles. The waiting time for this test was 24 hours, and the fracture width was 3.0 mm.

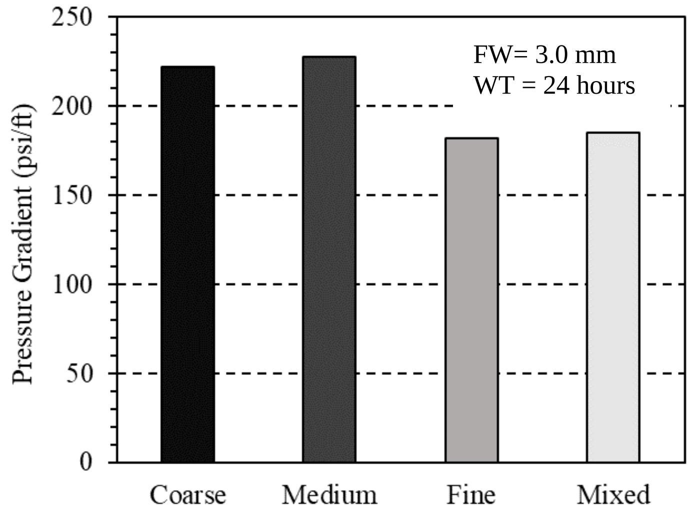

Figure 16. Sealing pressure for different mica particle size

The walnut shell particles were added slowly to the sample during the mixing to avoid flocculation. Figure 17 shows the sealing pressure for each concentration. The results showed that adding walnut shells to the sample improved its plugging efficiency and sealing ability. The sealing pressure increased from 183.04 to 392.76 psi/ft when adding 0.50% of walnut shells and 1104.97 psi/ft when adding 0.75% of walnut shells. Interestingly, adding 1.00% of walnut shells increased its ability to withstand high pressure significantly as we reached the limit pressure for our equipment, 5685 psi/ft, and we could not obtain breakthrough pressure. Since we could not reach the breakthrough pressure, there is no post-injection for the sample with 1.00% of walnut shells. The permeability decreased significantly after using the walnut shell to 3.079 and 2.538 md when 0.50 and 0.75% of walnut shells were added. Figure 18 shows the Frr results for 0.50 and 0.75% samples at different flow rates.

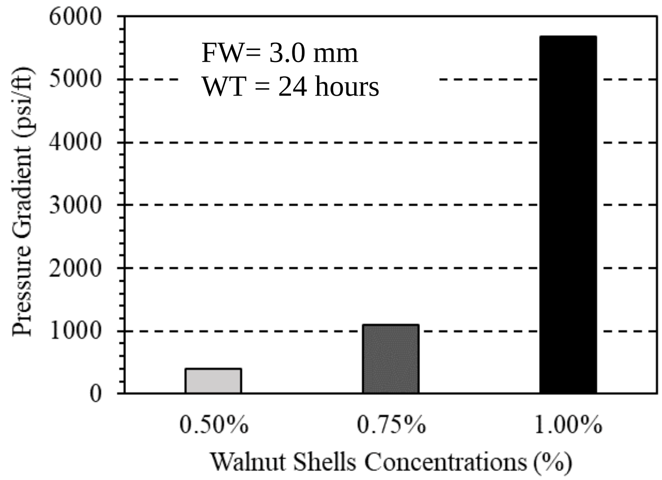

Figure 17. Sealing pressure for different walnut shell concentrations

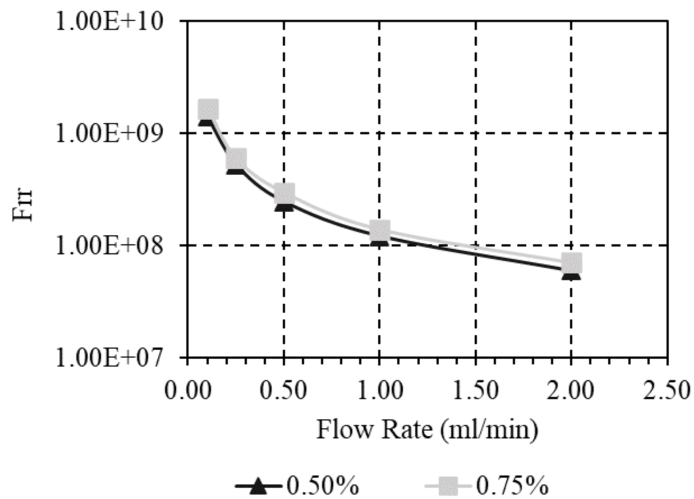

Figure 18. Frr results at different flow rates

5.4.4. Drilling Fluid Effect. Core flooding experiments were conducted using two samples, with and without drilling fluid, to better understand the impact of the drilling fluid on the sealing pressure. Figure 19 shows the sealing pressure of both samples. These experiments were conducted using a fractured cement core with a fracture width of 3 mm. The first sample was prepared by adding the LT-RPPG particles to the brine with a swelling ratio of 10, and this was then mixed. The second sample was prepared by adding the LT-

RPPG particles to the drilling fluid and then mixing. The obtained results showed that introducing the drilling fluid to the mixture results in higher sealing efficiency as it was 183 psi/ft when no drilling fluid was present compared to 290 psi/ft when drilling fluid was introduced. These results indicate that mixing the LT-RPPG with drilling fluid increases the plugging efficacy and the ability to withstand higher pressure. Figure 20 shows the Frr results at different flow rates.

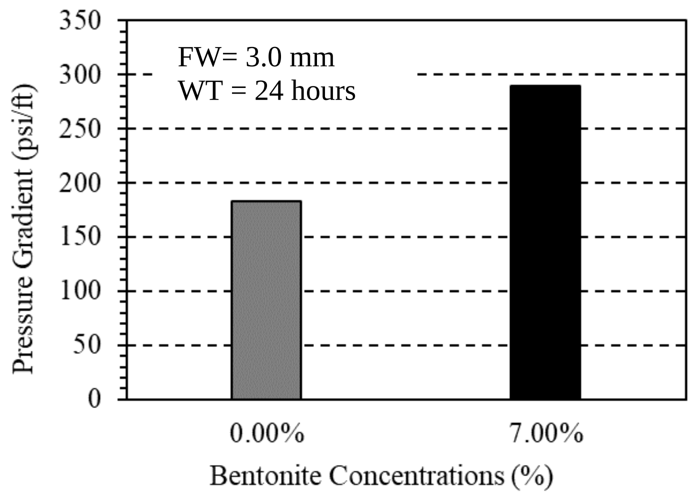

Figure 19. The sealing pressures in the presence and absence of bentonite

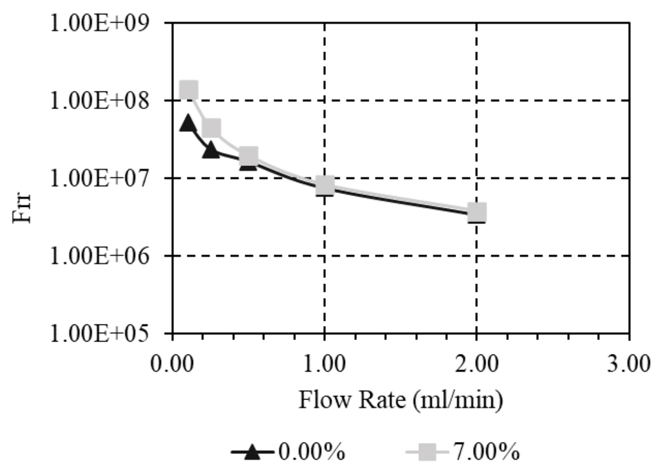

Figure 20. Frr results at different flow rates

5.4.5. Comparing LT-RPPG Sealing Efficiency to Conventional LCMs. In this study, the sealing pressure and plugging efficiency were investigated by adding different additives to the LT-RPPG. These additives included bentonite, mica, and walnut shell. The results demonstrated the impact of these additives on the LT-RPPG. Table 4 shows the results of sealing pressure and permeability for all the experiments. We found that the LT-RPPG sealing pressure could be significantly increased when adding 1% walnut shells. More experiments were conducted using different samples to show the importance of including the LT-RPPG in the mixture. Figure 21 shows the sealing pressures for different samples. Since mica is considered an LCM material, one of the experiments was conducted using drilling fluid mixed with mica to test its plugging efficiency compared to the LT-RPPG. The obtained results showed that the mixture of drilling fluid with mica provides very low sealing pressure. In contrast, when the LT-RPPG was added to the sample, the sealing pressure increased significantly to 222.24 psi/ft. Additionally, when the LT-RPPG with mica was tested, the sealing pressure was higher than it was with the drilling fluid mixed with mica as it was 136.76 psi/ft compared to 8.34 psi/ft. The results proved the importance of using the LT-RPPG to cure the loss as adding LT-RPPG to the mixtures increased the sealing pressure significantly.

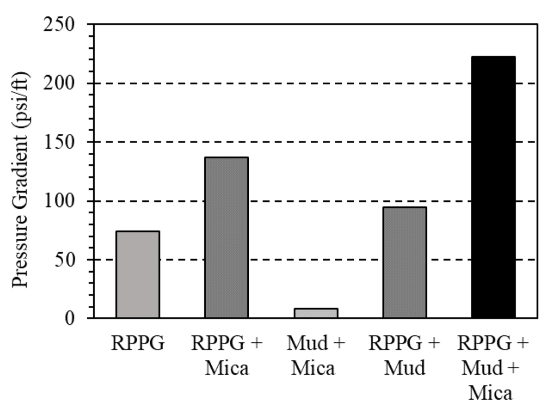

Figure 21. The sealing pressures for different samples

Table 4. Sealing pressure and permeability results for fracture width of 3.0 mm

Group	LT-RPPG Ratio	Bentonite Con. (%)	Mica Con. (%)	Mica Type	WS Con. (%)	WS Type	Sealing Pressure (Psi/ft)	Permeability (Darcy)	
								Before Treatment	After Treatment
#1	1:16	0	0	-	0	-	98.82	$7.59{\times}10^5$	0.1683
#1	1:16	7	0.50	C	0	-	151.36	$7.59{\times}10^5$	0.0129
#1	1:16	7	0.75	C	0	-	176.37	$7.59{\times}10^5$	0.0060
#1	1:16	7	1.00	C	0	-	222.24	$7.59{\times}10^5$	0.00567
#1	1:16	7	2.00	C	0	-	357.75	$7.59{\times}10^5$	0.00561
#1	1:16	7	0.50	Each	0	-	185.13	$7.59{\times}10^5$	0.0148
#1	1:16	7	1.00	M	0	-	227.66	$7.59{\times}10^5$	0.0052
#1	1:16	7	1.00	F	0	-	182.21	$7.59{\times}10^5$	0.0202
#2	1:10	0	0	-	0	-	183.04	$7.59{\times}10^5$	0.0459
#2	1:10	7	0	-	0	-	290.04	$7.59{\times}10^5$	0.0388
#2	1:10	0	0	-	0.50	F	392.76	$7.59{\times}10^5$	0.0031
#2	1:10	0	0	-	0.75	F	1104.97	$7.59{\times}10^5$	0.0025
#2	1:10	0	0	-	1.00	F	5685.41*	$7.59{\times}10^5$	-
#3	1:8	0	0	-	0	-	374.43	$7.59{\times}10^5$	0.0421
#4	1:5	0	0	-	0	-	1074.91	$7.59{\times}10^5$	0.0337
#5	-	7	1.00	C	0	-	8.34	$7.59{\times}10^5$	-
#6	-	7	0	-	1.00	F			

* C = Coarse, M= medium, F= fine.

6. CONCLUSIONS

The impact of different additives on LT-RPPG strength and plugging efficiency was evaluated utilizing rheology measurement and core flooding experiments. A summary of the findings deduced from this research is as follows:

- Using drilling fluids instead of brine to prepare LT-RPPG can increase recrosslinked bulk gel strength, its sealing pressure as well as plugging efficiency to open fractures.
- Adding mica to the mixture of LT-RPPG and drilling mud resulted in more robust plugging materials; however, increasing the mica concentration from 1.00 to 2.00%

did not significantly affect the gel strength but greatly increased the sealing pressure from 222.24 psi/ft to 357.75 psi/ft.

- The sealing pressure increased slightly when coarse mica was used compared to fine mica as it was 222.24 psi/ft when course mica was used compared to 182.21 psi/ft when fine mica was used.
- The fracture permeability was reduced more than 10^7 times for all experiments.
- Walnut shell improved the sealing pressure significantly, especially when 1.00% was added as it reached 5685 psi/ft and no breakthrough was occurred.

NOMENCLATURE

LT-RPPG	Low Temperature Re-Assembling Preformed Particle Gel
KCl	Potassium Chloride
SEM	Scanning Electron Microscope
Frr	Residual Resistance Factor
HPAM	Hydrolyzed Polyacrylamide Polymer
PEI	Polyethleneimine
SMP	Shape Memory Polymer
HMP Gel	Cross-linked Modified Polyacrylamide Gel
SR	Swelling Ratio
LCMs	Lost Circulation Materials
WS	Walnut Shell
FW	Fracture Width
WT	Waiting Time

ACKNOWLEDGEMENTS

The authors would like to thank the JIP members, including ConocoPhillips, Occidental Petroleum, and Daqing Wantong Chemical Plant, for their financial support.

REFERENCES

Ahdaya, M., & Imqam, A. (2019). Investigating geopolymer cement performance in presence of water based drilling fluid. Journal of Petroleum Science and Engineering, 176, 934-942. https://doi.org/10.1016/j.petrol.2019.02.010

Ahdaya, M., Al BRahim, A., Bai, B. et al. 2022. Low-Temperature Recrosslinkable Preformed Particle Gel as a Material for Lost Circulation Control. SPE J. SPE-209783-PA (in press; posted 21 March 2022). https://doi.org/10.2118/209783-PA.

Al Maskary S, Abdul Halim A, Al Menhali S, (2014): Curing losses while drilling & cementing.Abu Dhabi International Petroleum Exhibition and Conference, Abu Dhabi, UAE; 10-13November, SPE Paper# 171910. doi: https://doi.org/10.2118/171910-MS

Alsaba, M., Nygaard, R., Hareland, G., & Contreras, O. (2014, April). Review of lost circulation materials and treatments with an updated classification. In AADE National Technical Conference and Exhibition, Houston, TX (pp. 1-9).

Bao, D., Qiu, Z., Zhao, X., Zhong, H., Chen, J., & Liu, J. (2019). Experimental investigation of sealing ability of lost circulation materials using the test apparatus with long fracture slot. Journal of Petroleum Science and Engineering, 183, 106396. https://doi.org/10.1016/-j.petrol.2019.106396

Borling, D., Chan, K., Hughes, T., & Sydnask, R. (1994). Pushing out the oil with conformance control. Oilfield Review;(Netherlands), 6(2).

Cook, J., Growcock, F., Guo, Q., Hodder, M., Van Oort, E., 2011. Stabilizing the wellbore to prevent lost circulation. Oilfield Rev. 23.

Cui, K., Jiang, G., Xie, C., Yang, L., He, Y., Shen, X., & Wang, X. (2021). A novel temperature-sensitive expandable lost circulation material based on shape memory epoxy foams to prevent losses in geothermal drilling. Geothermics, 95, 102145.

Du, G., Peng, Y., Pei, Y., Zhao, L., Wen, Z., & Hu, Z. (2017). Thermo-responsive temporary plugging agent based on multiple phase transition supramolecular gel. Energy & Fuels, 31(9), 9283-9289.

Hashmat, M. D., Sultan, Abdullah S., Rahman, Saifur , and S. M. Hussain. "Crosslinked Polymeric Gels as Loss Circulation Materials: An Experimental Study." Paper presented at the SPE Kingdom of Saudi Arabia Annual Technical Symposium and Exhibition, Dammam, Saudi Arabia, April 2016. doi: https://doi.org/10.2118/182740-MS

Hinkebein, T. E., Behr, V. L., & Wilde, S. L. (1983). Static slot testing of conventional lost circulation materials (No. SAND-82-1080). Sandia National Labs., Albuquerque, NM (USA).

Howard, G.C., Scott, P.P., 1951. An analysis and the control of lost circulation. In: Annual Meeting of the ATME, Missouri, 19-21 February

Kumar, A., Savari, S., Whitfill, D., Jamison, D.E., 2010. Wellbore strengthening: the less studied properties of lost-circulation materials. In: SPE Annual Technical Conference and Exhibition, Florence, Italy, 19-22 September.

Lashkari, R., Tabatabaei-Nezhad, S. A., & Husein, M. M. (2021). Shape Memory Polyurethane as a Drilling Fluid Lost Circulation Material. Macromolecular Materials and Engineering, 306(11), 2100354.

Li, J., Qiu, Z., Zhao, X., Zhong, H., Yang, Y., & Huang, W. (2020). Effect of different parameters on sealing performance of lost circulation process with dynamic fracture aperture testing apparatus. Journal of Petroleum Science and Engineering, 193, 107357.

Moore, P.L.: Drilling Practices Manual, 1986. Second Edition. PennWell Publishing Company

Nayberg, T.M., and B.R. Petty. "Laboratory Study of Lost Circulation Materials for Use in Oil-Base Drilling Muds." Paper presented at the SPE Deep Drilling and Production Symposium, Amarillo, Texas, April 1986. doi: https://doi.org/10.2118/14995-MS

Pu, Jingyang, Bai, Baojun, Alhuraishawy, Ali, Schuman, Thomas, Chen, Yashu, and Xindi Sun. "A Recrosslinkable Preformed Particle Gel for Conformance Control in Heterogeneous Reservoirs Containing Linear-Flow Features." SPE J. 24 (2019): 1714–1725. doi: https://doi.org/10.2118/191697-PA

Qiu, J., Bai, Y., Sun, J., Dai, L., Lei, S., & Liu, F. (2022). Experimental study on an oil-based polymer gel for lost circulation control in high-temperature fractured formation. Journal of Applied Polymer Science, 51763.

Ramasamy, Jothibasu , Amanullah, Md , and Mohammad K Al-Arfaj. "A Large Aperture Test Apparatus for Severe Lost Circulation Material Evaluation." Paper presented at the SPE Kingdom of Saudi Arabia Annual Technical Symposium and Exhibition, Dammam, Saudi Arabia, April 2018. doi: https://doi.org/10.2118/192227-MS

Sanders MW, Scorsone JT and Friedheim JE, (2010): High-fluid-loss, high-strength lostcirculation treatments. SPE Deepwater Drilling and Completions Conf., Galveston, Texas, USA;5-6 October, SPE Paper#135472

White, Robert J. "Lost-circulation Materials and their Evaluation." Paper presented at the Drilling and Production Practice, New York, New York, January 1956.

Whitfill, D., & Miller, M. (2008, April). Developing and testing lost circulation materials. In AADE fluids conference and exhibition, Houston, Texas, USA (pp. 8-9).

Xie, B., Ma, J., Wang, Y., Tchameni, A. P., Luo, M., & Wen, J. (2021). Enhanced hydrophobically modified polyacrylamide gel for lost circulation treatment in high temperature drilling. Journal of Molecular Liquids, 325, 115155.

Xu, C., Zhang, J., Kang, Y., You, L., Yan, X., Cui, K., & Lin, C. (2019, March). Investigation on the transport and capture behaviours of lost circulation material in fracture with rough surface. In International Petroleum Technology Conference. International Petroleum Technology Conference.

PAPER

III. RECROSSLINKABLE PREFORMED PARTICLE GEL AS A LOST CIRCULATION MATERIAL FOR MEDIUM-TEMPERATURE APPLICATIONS

ABSTRACT

Lost circulation is a pervasive problem that occurs during well drilling and is quite serious and costly. It may result in well control difficulties and, in some cases, complete loss of the well; in addition, it is time-consuming, which significantly increases the total operational costs. This paper introduces a novel medium-temperature recrosslinkable preformed article gel (MT-RPPG), which is a swelling polymer gel, for medium temperature application, 80 to 100 °C, to overcome this issue. The MT-RPPG is composed of swellable gel particles that can self-crosslink to form a strong bulk gel in loss zones. Core flooding experiments were conducted to measure the sealing pressure. Different factors were investigated, including the MT-RPPG swelling ratio, fracture widths, and contamination of drilling fluids. The results indicated that decreasing the MT-RPPG swelling ratio will increase its ability to withstand higher pressure as it increased significantly from 197.77 to 6234.75 psi/ft when the swelling ratio decreased from ten to eight. It was found that the contaminates in the MT-RPPG with the drilling fluid will not affect its plugging efficiency, as it was found that the sealing pressure increased slightly from 197.77 to 202.84 psi/ft when drilling fluid was introduced. Also, the permeability decreased more than 10^6 times.

1. INTRODUCTION

Lost circulation is a very common, serious problem that drilling teams can encounter during drilling oil or gas wells. It mainly occurs when drilling through fractured formations which make it easy for the drilling fluid to enter these fractures and not return to the surface. Usually, drilling operations induce these fractures and result in more serious situations (Moore, 1986). The lost circulation can be classified based on the return into seepage (<10 bbl/hr), partial (10-50 bbl/hr), severe (>50 bbl/hr), or complete (total) loss (no return) (Nayberg & Petty, 1986; Ghalambor et al., 2014). Losing the return can be very costly since it can result in the loss of well control, which, in some cases, causes blowout. Moreover, lost circulation contributes significantly to increasing non-productive time (NPT), which leads to an enormous increase in the total cost of the drilling operation (Howard and Scott, 1951; Kumar et al., 2010). According to Wang et al. (2007) and Rehm et al. (2013), lost circulation issues contributed to more than 12% of the NPT in the Gulf of Mexico. Lost circulation results in an annual cost of one to four billion dollars (Ivan et al., 2003; Cook et al., 2011; Mirabbasi et al., 2022).

For several decades, researchers have studied and evaluated different materials to mitigate this problem. Conventional lost circulation materials (LCM) have been widely used to prevent or mitigate the losses, especially in minor fractures; however, they do not have a strong plugging ability due to the weak interaction between their particles (Jeennakorn, 2017; Yang, et al., 2022). Researchers have also studied the in-situ polyacrylamide-based polymer gels that are crosslinked by metallic ions, and it was found that they can succeed in preventing and plugging the loss zones; nevertheless, high salinity and high temperature can significantly impact their performance (Syed et al., 2014; Sun et

al., 2020; Yang et al., 2022). In addition, metallic crosslinkers are considered harmful and toxic to the environment. Further, several researchers investigated different materials, such as swellable materials, to mitigate losses during drilling through fractured formations. When these materials encounter formation water, drilling fluids, or chemical reagents, they will be activated, and form sealing plugs to stop or reduce the losses to the formations. Moreover, finding a material that is stable at high temperatures (80 to 100 °C) is a challenge. A few researchers have proposed a material that can handle these conditions. Gamage et al. (2014) investigated a fluid loss pill composed of water-soluble polymer and a metal-based crosslinker for high-temperature environments up to 140 °C; however, the gelation time of one hour was quick thus limiting its application. Lashkari et al. (2021) studied a shape memory polyurethane for lost circulation applications. They found that shape memory polyurethane can seal wide fractures at 80°C reservoirs after being activated by the formation temperature. Bai et al. (2022) studied the ability of polymer gel to seal fractured formations at 160 °C utilizing a core flooding test using fractured cores, and based on their finding, polymer gel can control loss in fractures.

Recently, recrosslinkable preformed particle gel (RPPG), which is a swellable polymer gel material, has been comprehensively investigated for conformance control applications (Pu et al., 2019; Long et al., 2020; Song et al., 2022a; Bai et al., 2022; Yu et al., 2022; Long et al., 2022; Song et al., 2022). RPPG has been applied in the West Sak field in Alaska, and it successfully controlled the fluid conduit issues with an improvement rate of 23% compared to traditional PPG (Targac et al., 2020). RPPG has many advantages, including good mechanical strength and thermal stability, no dilution by formation water and drilling fluids, adjustable particle sizes, controllable recrosslinking time, good

placement in fractures, and strong plugging efficiency with fractures since it can form a strong, bulky gel at target zones (Pu et al., 2019; Pu et al., 2021). These features make it a strong candidate to mitigate the lost circulation problems. Furthermore, Ahdaya et al. (2022) investigated the ability of using recrosslinkable preformed particle gel (RPPG) as a lost circulation material at low-temperature conditions. They found that the sealing pressure can reach up to 1381 psi/ft in fracture widths of 2.0 mm when smaller swelling ratios were used. However, this can only work for reservoir temperatures up to 80 °C.

In this paper, a novel MT-RPPG that can be used at medium temperature reservoirs, 80 to 100 °C, will be introduced. This MT-RPPG will be comprehensively evaluated to investigate its capability to control the severe losses during drilling operations at temperatures of 80 to 100 °C.

2. EXPERIMENTAL MATERIALS DESCRIPTION

MT-RPPG. MT-RPPG is a novel recrosslinkable performed particle gel that is composed of acrylamide, 2-acrylamide-2-methylpropane sulfonic acid, xanthan gum, Methylenebisacrylamide, and a pre-embedded Cr (III)-based crosslinker.

$$W_{brine} = (SR - 1) * Wt_{RPPG} \qquad (1)$$

Where SR is the swelling ratio, W_{brine} is the mass of water in grams, and Wt_{RPPG} is the RPPG mass in grams.

Figure 1 shows the MT-RPPG sample at different stages: (a) dry particles, (b) initial slurry, and (c) fully swollen particles. To prepare the MT-RPPG slurry, first, we gradually added the MT-RPPG particles to the brine and mixed until the particles became fully swelling. The time required for the MT-RPPG particles to become fully swelled was

between 30 minutes to 4 hours, depending on the desired swelling ratio since larger swelling ratios required more time for MT-RPPG to absorb the brine.

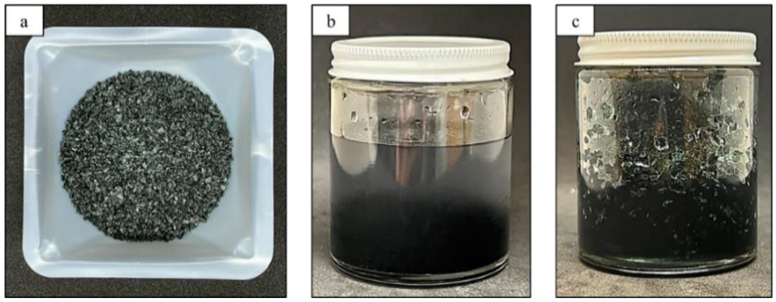

Figure 1. MT-RPPG sample preparation (a) MT-RPPG dry particles. (b) initial slurry. (c) fully swollen.

Brine. A solution of 2% potassium chloride (KCL) was used as a base fluid for all samples.

Drilling fluid. A basic formula of water base mud that contains 7% bentonite was used.

Cement cores. The fractured cemented cores were prepared by mixing tap water with cement powder which was then poured into the cylinder molds. A steel bar was placed in the center of the mold to create the desired fracture. The steel bar was removed 6 to 8 hours after the cement slurry was prepared and before the cement was fully set. Different fracture widths were used in this study, including 1.5 mm, 2 mm, and 3.0 mm.

HAAKE rheometer. A HAAKE rheometer was used in this study to measure the elastic modulus (G') and viscous modulus (G'') of each sample to better understand the effect of each parameter on the material's strength.

Scanning electron microscopy (SEM). The microstructure of the MT-RPPG was characterized with the HELIOS NANO LAB 600.

3. EXPERIMENTAL SETUP DESCRIPTION

The setup schematic design that was used in core flooding experiments is shown in Figure 2. The setup is composed of two accumulators, which are used for MT-RPPG and drilling fluid injections, a syringe pump to apply the injection flow rates, a core holder to hold the fractured cement core, and a pressure transducer and a computer to observe and record the pressure data during the experiment.

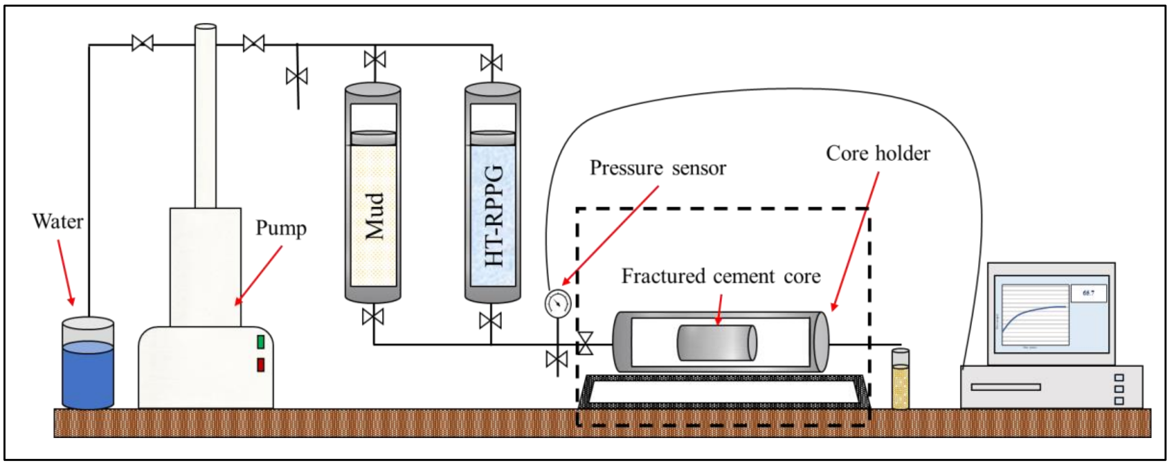

Figure 2. Core flooding setup design

4. EXPERIMENTAL PROCEDURE DESCRIPTION

The sealing pressure, which represents the highest pressure the material can hold before its failure, and residual resistance factor (Frr), which represents the plugging efficiency, were measured using core flooding experiments. The experiments were conducted using the following procedures. Initially, the fractured cement core was placed inside the core holder, and a confined pressure was applied. Then, the swollen MT-RPPG

sample was injected into the fracture using a syringe pump. The injection rate was 1.0 mL/min and was stopped after the injection pressure was stabilized. Then, the fractured cement core was placed in an oven at 100°C for 30 hours. After that, the fractured cement core was placed again in the core holder, and drilling fluid was injected using a flow rate of 1.0 mL/min until a breakthrough occurred. The pressure at which the breakthrough occurred is called the sealing pressure. Then, the post-injection took place, using 2% KCL solutions to obtain the permeability and Frr at different flow rates of 0.10, 0.25, 0.50, 1.00, and 2.00 mL/min. The permeability was calculated using the Darcy equation:

$$k = \frac{q\,\mu\,L}{A\,\Delta P} \quad \text{……………………………………………………(2)}$$

Where K is permeability, md, Q is flow rate, cc/sec, μ is viscosity, cp, L is core length, cm, A is core cross-section's area, cm^2, and ΔP is differential pressure, atm.

Then, the Frr was calculated using the following equation:

$$F_{rr} = \frac{K_{before}}{K_{after}} \quad \text{……………………………………………………(3)}$$

Where K_{before} is the permeability before treatment, and K_{after} is the permeability after treatment.

5. RESULTS

5.1. EFFECT OF SWELLING RATIO ON GEL STRENGTH

The swelling ratio is an important factor that affects the strength of the MT-RPPG. In this research, different MT-RPPG swelling ratios were used including 5, 8, 10, and 16. Table 1 shows the samples of MT-RPPG. The samples were prepared as follows: First, the 2% KCl solution was poured into the beaker and started the mixing using 300 rpm. After

that, the MT-RPPG particles were gradually added to the beaker while mixing to avoid particle aggregations. The mixture then was left to mix until the particles absorbed all of the solution. Then, the samples were placed in a high-temperature oven, 100°C, for 30 hours. After that, the samples were taken out of the oven, and G' and G" were measured. Figure 3 shows the results of G' and G'' for MT-RPPG with different swelling ratios. These results indicated, as anticipated, that the strength of the materials is strongly dependent on the concentrations of the MT-RPPG particles inside the mixture. The G' increased from 299.49 Pa to 647.95 Pa when the swelling ratio decreased from 16 to 10; however, it increased dramatically from 647.95 Pa to 4615.87 Pa when the swelling ratio decreased from 10 to 8. Figure 4 illustrates pictures that were taken for sample 3 (a) immediately after mixing, (b) after 1 hour at 100°C, (c) after 24 hours at 100°C, and (d) after 48 hours at 100°C. Figure 5 shows the SEM image for MT-RPPG with a swelling ratio of ten after fully recrosslinking. The image illustrates that the pores are interconnected and have strong bonds and thick walls as shown by the red arrows.

Table 1. MT-RPPG samples

Sample #	MT-RPPG Swelling Ratio	Brine Concentrations (%)
Sample 1	5	2 % KCl
Sample 2	8	
Sample 3	10	
Sample 4	16	

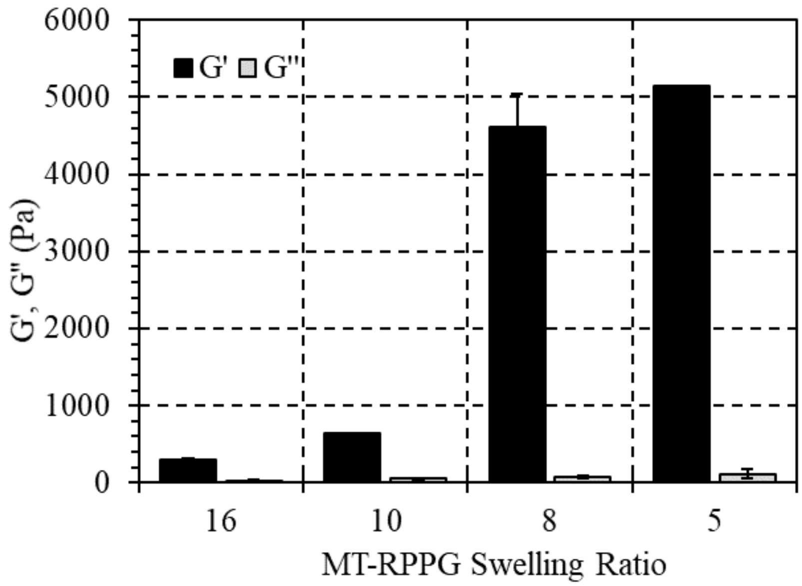

Figure 3. The G’ and G” results for MT-RPPG with different swelling ratios

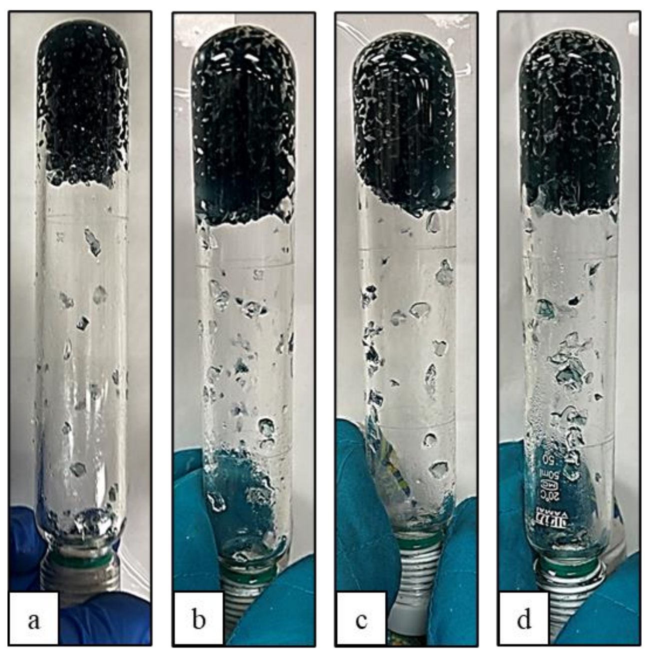

Figure 4. Pictures of sample 3 after different time. (a) immediately after mixing. (b) after 1 hour. (c) after 24 hours. (d) after 48 hours.

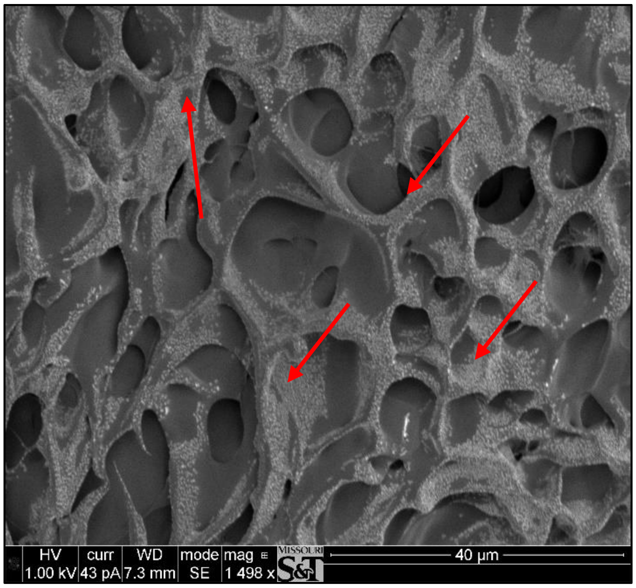

Figure 5. SEM image for sample 3 after fully recrosslinked.

5.2. CORE FLOODING EXPERIMENTS RESULTS

In order to evaluate the plugging performance of MT-RPPG, a series of experiments were conducted to study the influence of various factors on sealing pressure. Figure 6 shows an experiment result for sample 3. The fracture width for this experiment was 3.0 mm, the waiting time was 30 hours, and the temperature was 100°C. The MT-RPPG injection pressure increased for the first 30 minutes and then stabilized at 100 psi/ft. The MT-RPPG injection stopped after the pressure stabilized. Then the fractured cement core was placed in an oven at 100°C for 30 hours. After that, drilling fluid was injected, using a flow rate of 1 mL/min. The breakthrough pressure (sealing pressure) was 197.77 psi/ft.

After the experiments were concluded, the core was acquired and broken to analyze the remaining gel inside. Figure 7 shows the fractured cement core before and after the experiment. Figure 8 shows a picture of the remaining gel inside the fracture. As illustrated by the picture, some gel remained in the fracture even after the post-injection, which played

a critical role in decreasing the permeability more than 10^6 times from 2. 85E+07 md to 2.84 md. Figure 9 shows the Frr results for this experiment at different flow rates.

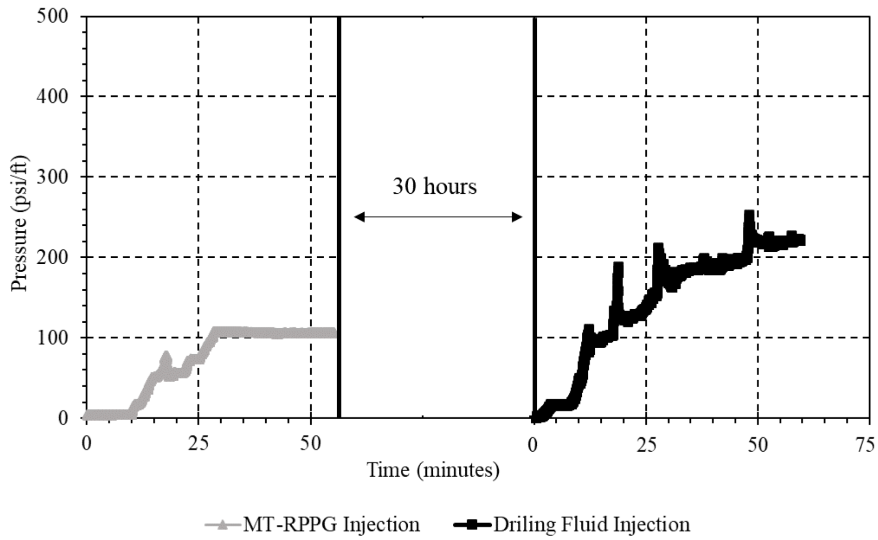

Figure 6. Core flooding results for sample 3

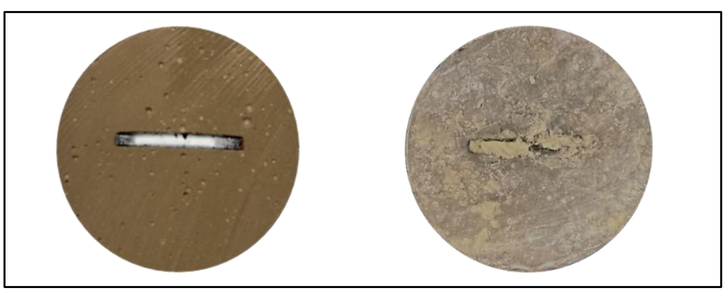

Figure 7. Fractured cement core before and after the experiment

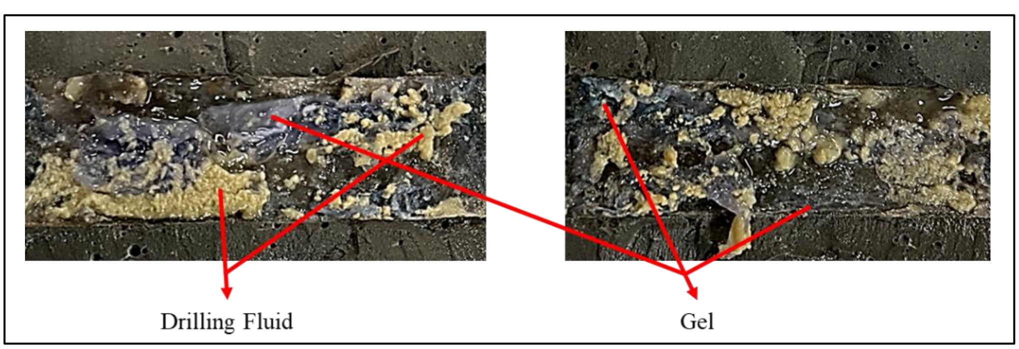

Figure 8. Image inside the fracture after the experiment

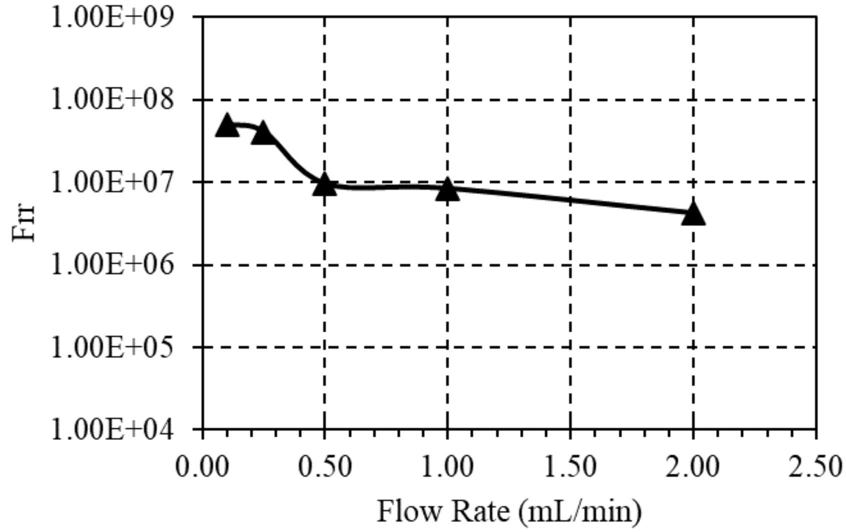

Figure 9. Frr results for sample 2 at different flow rates

5.2.1. Fracture Width Effect. Drilling fluid loss is mainly dependent on the size of fracture widths, which can significantly impact the return of the drilling fluid to the surface. Since it is hard to predict the fracture size and widths, evaluating a material that can plug different sizes is essential. Thus, in this research, different fracture widths were used, including 1.5, 2.0, and 3.0 mm. For all of these experiments, the base formula was MT-RPPG with a swelling ratio of 10. The waiting time was 30 hours, and the temperature was 100°C. The results of the sealing pressure for different fracture widths are shown in Figure 10. The MT-RPPG with this swelling ratio could withstand as high as 197.77 psi/ft when a fracture width of 3.0 mm was used. Using smaller fracture widths, such as 2.0 and 1.5 mm, resulted in higher sealing pressure of 883.69 and 927.3 psi/ft, respectively. It is always possible to reduce the swelling ratio to obtain higher sealing pressure and higher performance. The permeability decreased more than 106 times in all of the experiments. Figure 11 shows the Frr results for different fracture widths at different flow rates.

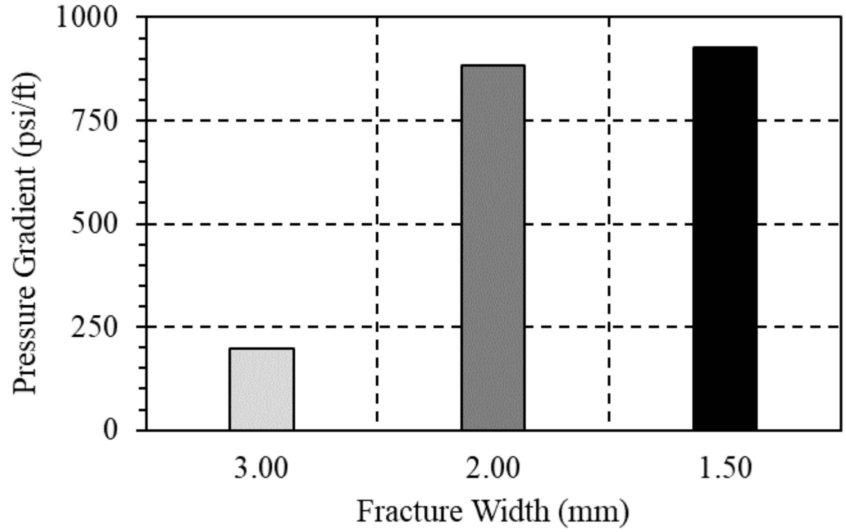

Figure 10. Sealing pressure at different fracture widths

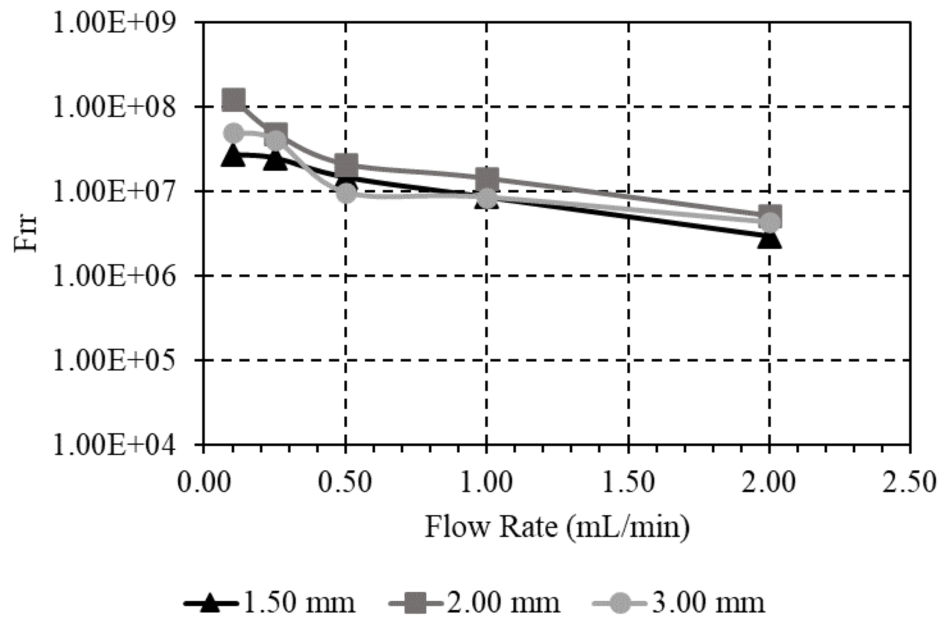

Figure 11. The Frr results for different fracture widths at different flow rates.

5.2.2. Swelling Ratio Effect. Swelling ratio plays a critical role in the strength of the material. As the swelling ratio decreases, the material gains more strength and will provide more plugging efficacy. In this research, three different swelling ratios were evaluated, including 16, 10, and 8. For all of these experiments, the waiting time was 30

hours, the temperature was 100°C, and the fracture width was 3.0 mm. Figure 12 illustrates the results of sealing pressure for MT-RPPG with different swelling ratios. It is very clear that the swelling ratios affected the sealing pressure, especially between swelling ratios ten and eight. The sealing pressure increased significantly to 6234.75 psi/ft (the limitation of our equipment) when a swelling ratio of eight was used compared to 197.77 psi/ft when a swelling ratio of ten was used. As mentioned previously, decreasing the swelling ratio allows a more robust material to form, which increases its ability to withstand higher pressure. Since the breakthrough could not be reached for a swelling ratio of eight, the permeability could not be measured. However, the permeability was measured for swelling ratios of 10 and 16, and it was found that the permeability was reduced more than 10^6 times for both experiments. Figure 13 shows Frr results for both swelling ratios (10 and 16) at different flow rates.

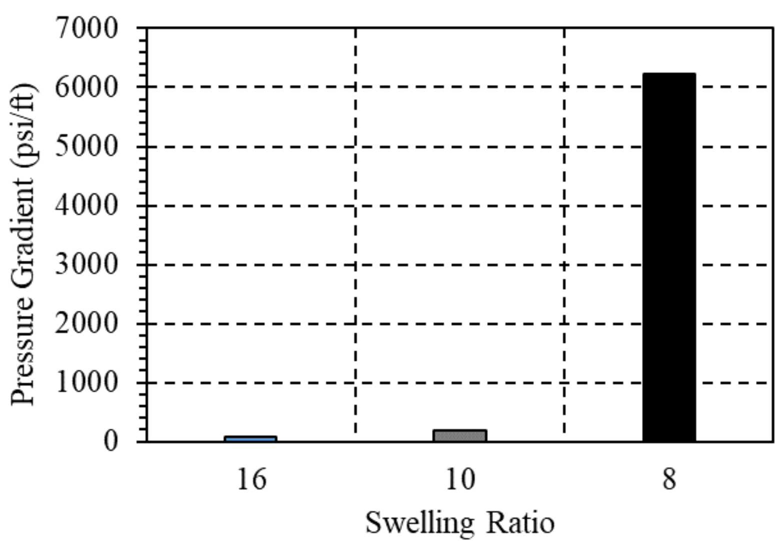

Figure 12. Sealing pressure at different swelling ratios

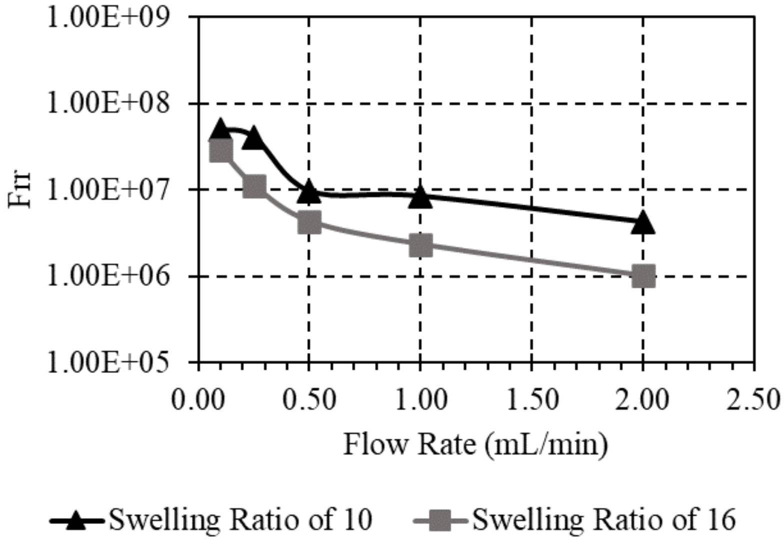

Figure 13. The Frr results for different swelling ratios at different flow rates

5.2.3. Waiting Time Effect. Since the non-productive time is one of the main issues that impact the drilling cost, the time at which the MT-RPPG required to fully function was investigated in this study. Different waiting times were investigated including 6, 30, and 60 hours. Three samples were prepared utilizing MT-RPPG with swelling ratio of ten and injected into the fractured cement core. After the injection was conducted, the fractured cement cores were placed in the oven at 100°C. After six hours the first core was tested to measure the sealing pressure after waiting time of six hours. After 30 and 60 hours, the second and the third cores were tested respectively to estimate the sealing pressure at each waiting time. The fracture cement core had a width of 3.0 mm. Figure 14 shows the sealing pressure of MT-RPPG at different waiting time. The sealing pressure was around 338.7 psi/ft when the waiting time was six hours, however it decreased to 197.77 psi/ft when the waiting time was 30 hours. This could be due to that the MT-RPPG after six hours is still particles and did not recrosslinked which resulted in higher sealing pressure. When, the waiting time was 60 hours the sealing pressure increased significantly to 718.44

psi/ft. This due to that the MT-RPPG could gain strength with time and form stronger plug. The permeability decreased more than 10^6 times since some amount of gel remained in the fracture.

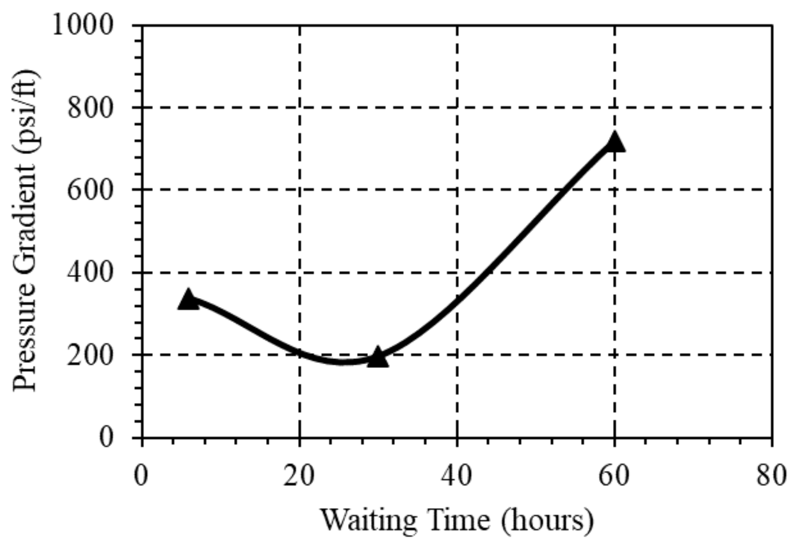

Figure 14. Sealing pressure after different waiting times

5.2.4. Drilling Fluid Effect. One possibility is for the LCMs to be contaminated with drilling fluid during the process of the treatment. Hence, evaluating the performance of the material in the presence of drilling fluid is critical. To study this effect, a sample of MT-RPPG with a swelling ratio of ten was prepared using a basic drilling fluid formula that contained 7.0% bentonite. The sample was prepared by adding the MT-RPPG particles to the drilling fluid while mixing for approximately 30 minutes. Figure 15 shows the G' and G'' results for the MT-RPPG sample with a ratio of ten in both the presence and absence of drilling fluid. The results indicate that the bentonite enhanced the strength of the material since it increased its G' from 647.95 Pa to 5160.97 Pa when the drilling fluid was introduced. This could be due to the fact that the bentonite absorbed some of the water,

which would reduce the actual MT-RPPG swelling ratio, resulting in higher strength. Figure 16 shows the SEM image for the MT-RPPG sample with drilling fluid. The SEM image shows that the sample still has strong bonds between the pores, as demonstrated by the red arrows. Furthermore, a core flooding test was conducted to measure the sealing pressure for the sample after being exposed to drilling fluid. Figure 17 shows the sealing pressure results for both samples (with and without drilling fluid). The sealing pressure results indicate that there was almost no impact of the drilling fluid on the sealing pressure as it increased its ability to hold pressure slightly when the drilling fluid was present. This indicates that the MT-RPPG could work properly as lost circulation material even if the drilling fluid is present. The result of permeability indicated a reduction of more than 10^6 times. Figure 18 shows the Frr results for MT-RPPG samples at different flow rates.

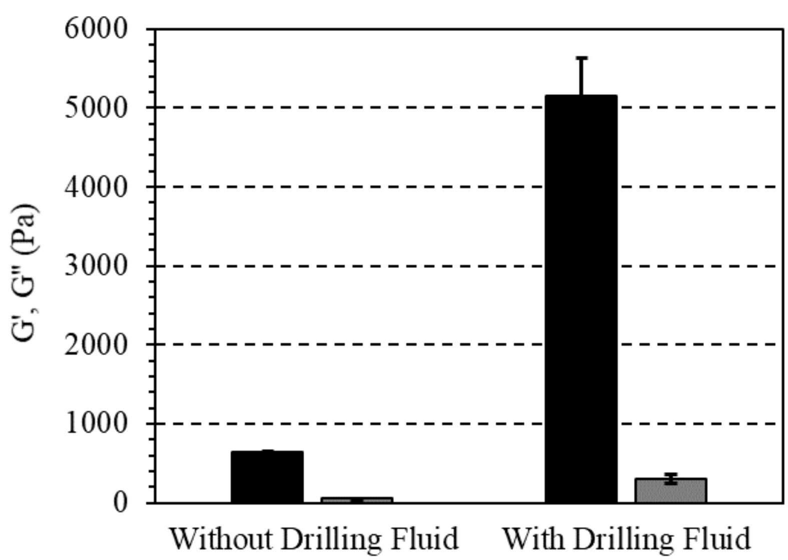

Figure 15. G’ and G” for MT-RPPG in the presence and absence of drilling fluid

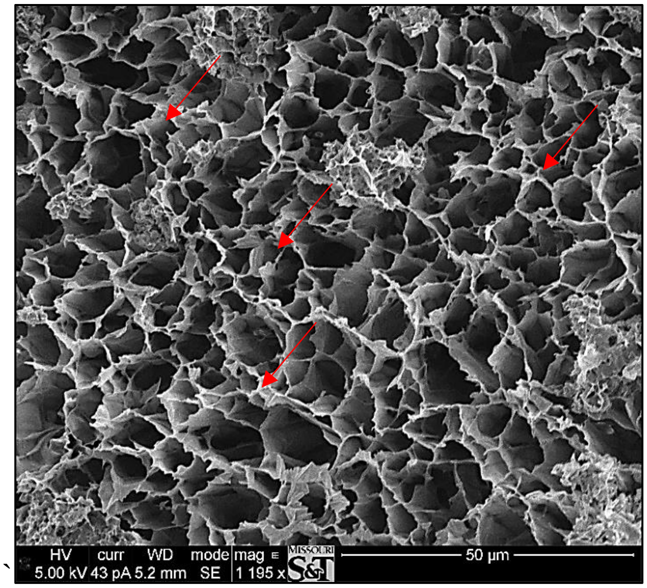

Figure 16. SEM image for MT-RPPG sample in the presence of drilling fluid

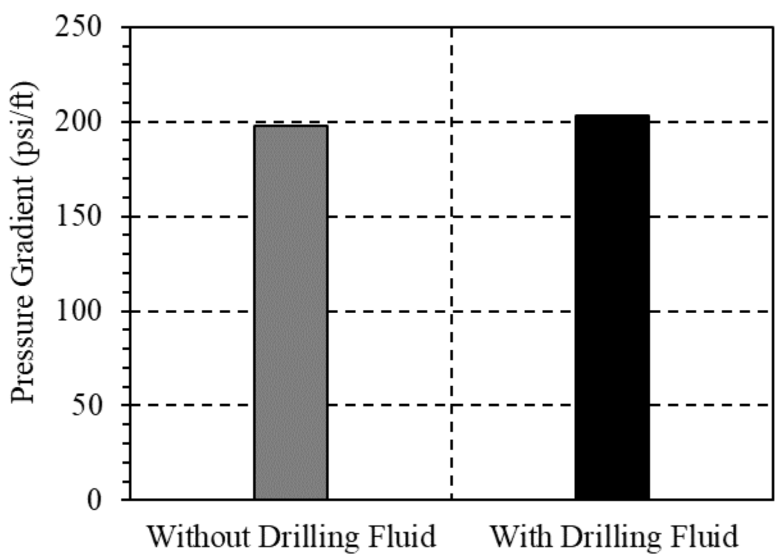

Figure 17. The sealing pressure results for MT-RPPG in the presence and absence of drilling fluid

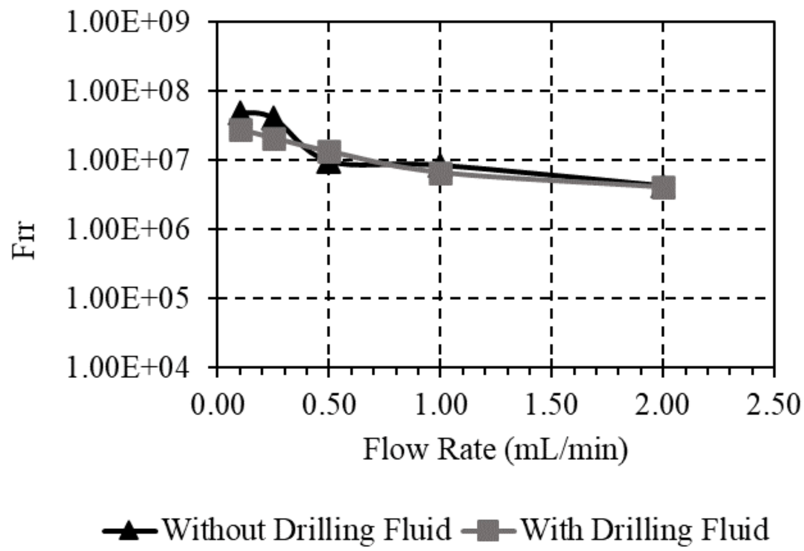

Figure 18. The Frr results for MT-RPPG samples in the presence and absence of drilling fluids at different flow rates

6. CONCLUSION

A MT-RPPG was evaluated as fluid loss control material in reservoirs with medium temperatures (80 to 100°C). The main factors investigated in this study were the effect of swelling ratios, fracture widths, drilling fluid contaminations on MT-RPPG strength, and plugging efficiency. The results were very optimistic since the MT-RPPG could withstand high pressure in high-temperature environments and reduced the permeability significantly even after it broke. A detailed summary of this research is as follows:

- A strong MT-RPPG mixture can be achieved when using higher particle concentrations, which provided higher strength materials with G’ as high as 5138.11 Pa for a swelling ratio of eight.

- Based on the results, the fracture width would not be an issue since the MT-RPPG can plug fracture widths from 1.5 mm up to 3.0 mm when using the proper swelling ratio.
- Waiting time has significant impact on sealing pressure as it increased from 197.77 to 718.44 psi/ft when waiting time was increased from 30 to 60 hours.
- Mixing the MT-RPPG with drilling fluid increased its strength significantly; however, it did not affect its ability to plug fractures.
- The remaining gel inside the fracture provided a high reduction in the permeability (more than 10^6 times), which indicates that even after the MT-RPPG reached its ability to withstand pressure, the reduction of the permeability significantly reduced the fluid loss.

ACKNOWLEDGEMENTS

The authors would like to thank the JIP members, including ConocoPhillips, Occidental Petroleum, and the Daqing Wantong Chemical Plant, for their financial support.

NOMENCLATURE

MT-RPPG	Medium temperature Re-crosslinkable Preformed Particle Gel
KCl	Potassium Chloride
SEM	Scanning Electron Microscope
Frr	Residual Resistance Factor
SR	Swelling ratio

REFERENCES

Ahdaya, Mohamed, Al Brahim, Ali, Bai, Baojun, and Thomas Schuman. "Low-Temperature Recrosslinkable Preformed Particle Gel as a Material for Lost Circulation Control." SPE J. (2022;): doi: https://doi.org/10.2118/209783-PA

Bai, B., Wang, Z., Sun, Z., Pu, J., & Schuman, T. (2022). U.S. Patent No. 11,268,009. Washington, DC: U.S. Patent and Trademark Office.

Bai, Y., Liu, C., Sun, J., Shang, X., Lv, K., Zhu, Y., & Wang, F. (2022). High Temperature Resistant Polymer Gel as Lost Circulation Material for Fractured Formation during Drilling. Colloids and Surfaces A: Physicochemical and Engineering Aspects, 128244.

Ghalambor, Ali , Salehi, Saeed , Shahri, Mojtaba P., and Moji Karimi. "Integrated Workflow for Lost Circulation Prediction." Paper presented at the SPE International Symposium and Exhibition on Formation Damage Control, Lafayette, Louisiana, USA, February 2014. doi: https://doi.org/10.2118/168123-MS

Ivan, C., Bruton, J., & Bloys, B. (2003). How can we best manage lost circulation. Radisson Astrodome Houston, Texas.

Jeennakorn, M. (2017). The effect of testing conditions on lost circulation materials' performance in simulated fractures. Missouri University of Science and Technology.

Lashkari, R., Tabatabaei-Nezhad, S. A., & Husein, M. M. (2021). Shape Memory Polyurethane as a Drilling Fluid Lost Circulation Material. Macromolecular Materials and Engineering, 306(11), 2100354.

Long, Y., Bai, B., & Schuman, T. P. (2020). Re-Crosslinking Particle Gel for Co_2 Conformance Control and CO_2 Leakage Blocking.

Long, Y., Bai, B., & Schuman, T. P. (2022). U.S. Patent No. 11,214,729. Washington, DC: U.S. Patent and Trademark Office.

Mirabbasi, S. M., Ameri, M. J., Alsaba, M., Karami, M., & Zargarbashi, A. (2022). The evolution of lost circulation prevention and mitigation based on wellbore strengthening theory: A review on experimental issues. Journal of Petroleum Science and Engineering, 110149.

Pu, J., Bai, B., & Schuman, T. (2021). U.S. Patent No. 11,162,016. Washington, DC: U.S. Patent and Trademark Office.

Pu, J., Bai, B., Alhuraishawy, A., Schuman, T., Chen, Y., & Sun, X. (2019). A recrosslinkable preformed particle gel for conformance control in heterogeneous reservoirs containing linear-flow features. SPE Journal, 24(04), 1714-1725. doi: https://doi.org/10.2118-/191697-PA

Rehm, B., Schubert, J., Haghshenas, A., Paknejad, A. S., & Hughes, J. (2013). Managed pressure drilling. Elsevier.

Song, T., Ahdaya, M., Zhao, S., Zhao, Y., Schuman, T., & Bai, B. (2022). Evaluation of a Novel Recrosslinkable Hyperbranched Preformed Particle Gel for the Conformance Control of High-Temperature Reservoirs with Fractures. SPE Journal, 1-13.

Song, T., Zhai, Z., Liu, J., Eriyagama, Y., Ahdaya, M., Alotibi, A., ... & Bai, B. (2022). Laboratory evaluation of a novel Self-healable polymer gel for CO2 leakage remediation during CO2 storage and CO2 flooding. Chemical Engineering Journal, 444, 136635.

Sun, X., Bai, B., Alhuraishawy, A. K., & Zhu, D. (2021). Understanding the plugging performance of HPAM-Cr (III) polymer gel for CO2 conformance control. SPE Journal, 26(05), 3109-3118.

Syed, A., Pantin, B., Durucan, S., Korre, A., & Shi, J. Q. (2014). The use of polymer-gel solutions for remediation of potential CO2 leakage from storage reservoirs. Energy Procedia, 63, 4638-4645.

Targac, G., Gallo, C., Smith, D., Huang, C. K., Autry, S., Peirce, J., & Baohong, L. (2020, October). Case History of Conformance Solutions for West Sak Wormhole/Void Space Conduit with a New Reassembling Pre-formed Particle Gel RPPG. In SPE Annual Technical Conference and Exhibition. OnePetro. doi: https://doi.org/10.2118/201302-MS

Wang, H., Towler, B. F., & Soliman, M. Y. (2007, February). Fractured wellbore stress analysis: sealing cracks to strengthen a wellbore. In SPE/IADC drilling conference. OnePetro.

Yang, L., Xie, C., Ao, T., Cui, K., Jiang, G., Bai, B., ... & Tian, W. (2022). Comprehensive evaluation of self-healing polyampholyte gel particles for the severe leakoff control of drilling fluids. Journal of Petroleum Science and Engineering, 212, 110249.

Yu, B., Zhao, S., Long, Y., Bai, B., & Schuman, T. (2022). Comprehensive evaluation of a high-temperature resistant re-crosslinkable preformed particle gel for water management. Fuel, 309, 122086.

PAPER

IV. HIGH-TEMPERATURE RECROSSLINKABLE PREFORMED PARTICLE GEL AS A MATERIAL FOR LOST CIRCULATION

ABSTRACT

Lost circulation has become one of the biggest challenges that drilling engineering faces during drilling, especially at high-temperature reservoirs. The consequences of lost circulation can vary from an economic aspect as well as a safety aspect. In this paper, the capability of a novel high-temperature recrosslinkable preformed particle gel (HT-RPPG) is evaluated to see whether it can be better used to control severe and total losses. The HT-RPPG is injected in form of dispersed swellable gel particles but it can self-crosslink to form strong bulk gel after being placed in target zones. The sealing pressure and plugging efficiency of the HT-RPPG were evaluated utilizing core flooding test. Various impacting factors were investigated, including the swelling ratios, fracture widths, and bentonite concentrations. Results indicated that HT-RPPG is an excellent material that can be used control the severe loss of drilling fluids in the fractured reservoirs with the temperature up to 130 °C. The recrosslinked RPPG could withstand pressure up to 1077 psi/ft for the fractures up to 2 mm, and permeability was reduced more than 10^7 times.

1. INTRODUCTION

Lost circulation is a phenomenon that occurs while drilling in fractured formations. This could begin with seepage loss lower than 10 bbl/hr, and could increase until none returns to the surface (Moore, 1986). The lost circulation problems are not limited to one area. These problems can be encountered at any depth when the pressure applied to the formation exceeds the formation breakdown pressure. The outcomes of these issues can result in a significant increase in operational costs since it will greatly increase the nonproductive time (NPT). Based on the literature, the annual estimation cost worldwide of lost circulation is one to four billion dollars (Sanders et al., 2010; Cook et al., 2011). According to the US Department of Energy, approximately 10-20% of the total expenses of drilling at high-temperature and high-pressure wells are due to lost circulation issues (Growcock et al., 2009). Moreover, these issues can result in well control issues that can lead to loss of the well or blowouts. Lost circulation treatment is classified into two categories based on the period at which they were introduced: preventive treatment, also known as wellbore strengthening, and corrective treatment (Kumar and Savari, 2011). Corrective treatments are usually added to the drilling fluid after the lost circulation has occurred (White, 1956). Usually, conventional LCMs are used to cure seepage and partial losses as their function is to plug permeable formations or minor fractures to prevent losses. Depending on their appearance or physical properties, LCMs are classified into fibrous, flaky, or granular materials or a blend of the three. Alsaba et al. (2014) reclassified LCMs into seven categories including granular, flaky, fibrous, a mixture of LCMs, acid-soluble/water-soluble, high-fluid loss LCMs squeeze, swellable/hydratable LCM combinations, and nanoparticles

The performance of LCMs has been evaluated utilizing two methods: a high-pressure high-temperature (HPHT) filter press or particle plugging apparatus (PPA) using slotted discs to measure the volume of the fluid loss and core flooding experiments to measure sealing efficiency (Kumar, and Savari, 2011; Van Oort et al., 2011). Hinkebein et al. (1983) studied different conventional LCMs at different temperatures. They found that the LCM could seal fractures of 1.52 mm at room temperature, but it failed at high temperatures (204.5°C). Lecolier et al. (2005) evaluated a nanocomposite gel as a lost circulation material for high-temperature 120°C reservoirs,. They claim that the nanocomposite gel effectively reduced the permeability of high-permeable formations. The gelation time at high-temperature reservoirs is one of the challenges. Gamage et al. (2014) investigated an LCM pill that consists of a water-soluble polymer and a metal-based crosslinker for high-temperature applications up to 176°C. The minimum gelling time ranged from one to five hours, depending on the temperature. Kulkarni et al. (2016) studied LCM pills for high-temperature applications. They used various particulate components with different carrier fluids and studied them at 121 and 162.7°C. They found that the plugging performance is highly dependent on carrier fluids at high temperatures. Etthadi and Altun (2017) investigated the stability of using calcium carbonate in sepiolite drilling fluid for high-temperature applications. They found that using sepiolite-based mud with calcium carbonate plugged the pores at temperatures up to193°C. Mansour et al. (2017) studied a shape memory polymer (SMP) that was activated by the temperature of 70°C, and they found that it can plug fractures of 2.54 mm. Deng et al. (2018) studied a cross-linked polyacrylamide gel. They used a microencapsulated initiator to control the gelation time. The temperature limitation for this gel was 90°C. Ab Lah et al. (2019) studied

eggshells at 49°C as lost circulation material utilizing an HTHP filter press. Their results indicated that the coarse size of eggshells obtained the best performance for plugging applications. Jiang et al. (2019) studied cross-linked polyacrylamide gel for lost circulation at high temperatures, 80 to 150°C. They mentioned that it could hold 1000 psi at 150°C when using a fracture width of 3.00 mm. Xie et al. (2021) evaluated cross-linked polyacrylamide gel (HMP gel) to cure lost circulations in a high-temperature environment utilizing a customized bridging material apparatus. They found that this gel withstood a pressure of 600 psi at 140°C when a fracture width of 3.0 mm was used. Cui et al. (2021) studied a shape memory polymer for high-temperature applications. They found that when it was activated at a temperature of 95°C, it withstood a pressure of 800.6 psi. Wang et al. (2021a) studied a supramolecular gel (GP-A) at high temperature, and they found that the GP-A gel has good thermal stability under 200°C, and it could be used in fractured formations to reduce fluid losses. Wang et al. (2021b) conducted studies to investigate polymer gel plugging efficiency, and they found the plugging can reach up to 154.2 Kpa (22.36 psi) when the fracture width was 1.2 mm. Sonmez et al. (2021) studied calcium carbonate as a lost circulation additive at 148°C. The highest plugging performance was obtained when adding 50 lb/bbl of $CaCO_3$. Bai et al. (2022) studied a polymer gel for high-temperature applications up to 160°C. Their results showed a sealing pressure of 613.5 psi for a fracture width of 1.00 mm and 601.9 psi for a fracture width of 3.00 mm.

The lost circulation has been addressed by many researchers yet is still a major challenge, especially in high-temperature reservoirs (Du et al., 2017). In our previous research (Ahdaya et al., 2022), a low-temperature RPPG was evaluated and proved its success at plugging fractures at low temperatures, up to 80°C. This paper will introduce

the high-temperature RPPG to be evaluated as a lost circulation material for high-temperature applications, 130 °C.

2. EXPERIMENTAL MATERIALS DESCRIPTION

High-temperature recrosslinkable preformed particle gel. The HT-RPPG used in this study is yellow granular dry particles consisting of crosslinked poly (acrylamide-co-N-vinyl-2-pyrrolidone) (Yu et al., 2022). Swelling RPPG with pre-determined swelling ratios were prepared by gradually adding the HT-RPPG particles to the brine to avoid flocculation. The particle size range was 1 - 4 mm.

Brine. A solution composed of 2% potassium chloride (KCL) was utilized in this study.

Drilling fluid. Water-based mud with 7% bentonite was used in this study. The drilling fluid was prepared using a Hamilton Beach mixer, mixed for at least 10 minutes, and left overnight for bentonite prehydration to ensure that the bentonite was fully swelled (Ahdaya and Imqam, 2019).

Fractured cement cores. Several fracture widths were prepared including 1.5 mm, 2.0 mm, and 3.0 mm. The preparation of these cores was conducted by initially fixing a steel bar to the center of the cylinder mold to initiate the fracture within the cement core. Then, the cement paste was poured into the mold, and before the cement was fully set, the steel bar was removed to create a fracture.

HAAKE rheometer. The elastic modulus (G') and viscous modulus (G'') of each sample were measured using A HAAKE rheometer.

Scanning electron microscopy (SEM). The microstructure of the HT-RPPG was characterized with HELIOS NANO LAB 600.

3. EXPERIMENTAL SETUP DESCRIPTION

The experimental setup design is shown in Figure 1. The setup is composed of two accumulators, for sample and mud injection, a core holder to retain the fractured core, a pressure transducer to record the pressure response during the trial, and a syringe pump.

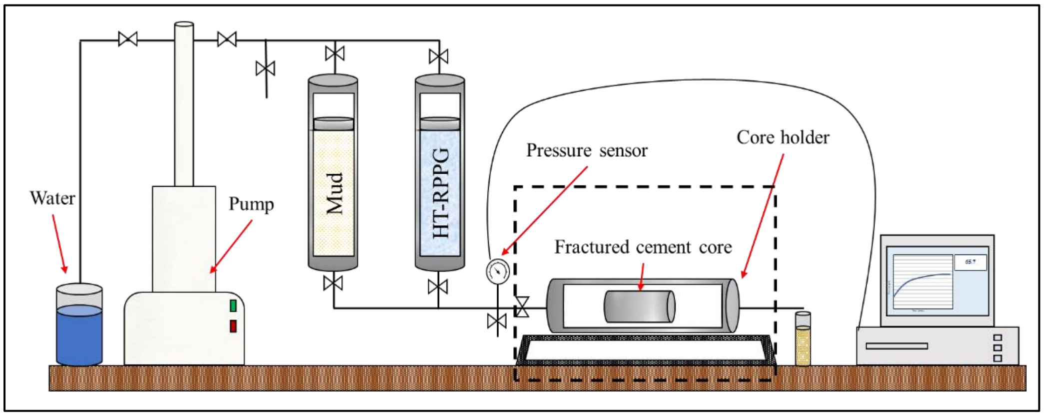

Figure 1. Core flooding setup design

4. EXPERIMENTAL PROCEDURE DESCRIPTION

The evaluation of sealing pressure, which is the highest pressure the material can withstand before it fails, and the residual resistance factor (Frr) was obtained utilizing core flooding experiments using fractured cement cores. Initially, the fractured core was placed inside the core holder, and confining pressure was applied. Then, the first accumulator was used to inject the pre-treatment drilling fluid injection into the fractured core to simulate

the actual scenario. After that, the second accumulator was used to inject the sample into the fractured core utilizing a flow rate of 1 cc/min until the pressure was stabilized. Next, the setup was disassembled to collect the core. Then, the fractured core was isolated and placed in a high-temperature vessel and placed at 130°C for 24 hours to allow the HT-RPPG to recrosslink. Subsequently, the core was placed again within the core holder, and post-injection using drilling fluid was conducted until a breakthrough was obtained.

After the sealing pressure was obtained, a post-injection was performed using 2% KCl brine to measure Frr. Various flow rates were used including 0.10, 0.25, 0.50, 1.00, and 2.00 cc/min. The permeability was calculated using the Darcy equation:

$$k = \frac{q\,\mu\,L}{A\,\Delta P} \quad \text{(1)}$$

Where K is permeability, md, Q is flow rate, cc/sec, μ is viscosity, cp, L is core length, cm, A is cross-sections area, cm^2, and ΔP is differential pressure, atm.

Then, Frr was calculated using the following equation:

$$F_{rr} = \frac{K_{before}}{K_{after}} \quad \text{(2)}$$

Where, K_{before} is the permeability before the treatment and K_{after} is the permeability after the treatment.

5. RESULTS AND DISCUSSION

5.1. EFFECT OF SWELLING RATIO ON GEL STRENGTH

In this research, different HT-RPPG swelling ratios were used including 5, 8, 10, 16, and 20. The swelling ratios were prepared using the following equation:

$$W_{brine} = (SR - 1) * Wt_{RPPG} \quad \text{(3)}$$

Where SR is the swelling ratio; W_{brine} is the mass of water in grams, and Wt_{RPPG} is the RPPG mass in grams.

Table 1 shows the samples used in this study. Figure 2 shows the HT-RPPG dry particles (a), the initial slurry (b), and fully swelled particles (c), separately. The sample was mixed using magnetic stirrer at low speed. The duration of mixing depends on the swelling ratios; it can range from 30 to 120 minutes.

Table 1. HT-RPPG samples

#	RPPG Ratio	Brine
Sample 1	1:20	2 % KCl
Sample 2	1:16	
Sample 3	1:10	
Sample 4	1:8	
Sample 5	1:5	

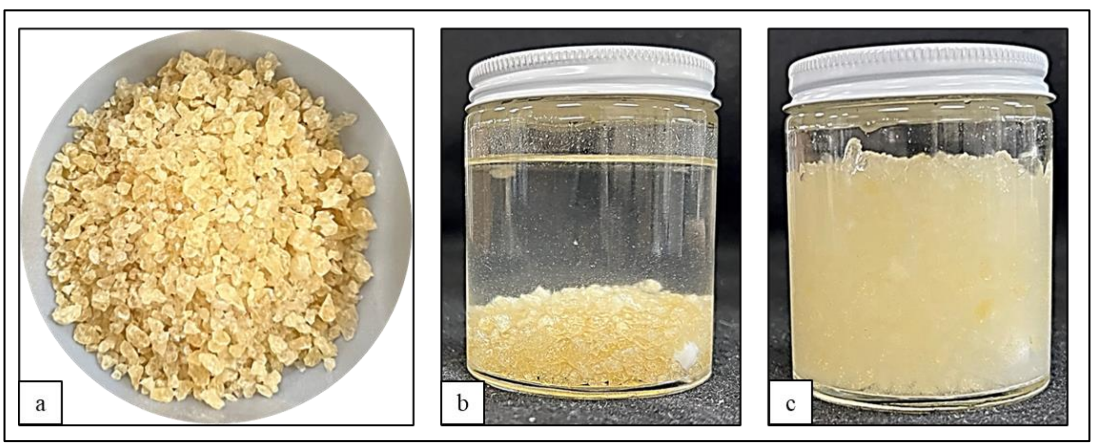

Figure 2. HT-RPPG sample (a) HT-RPPG dry particles. (b) initial slurry. (c) fully swelled.

Figure 3 shows the G’ and G” for samples 1, 2, 3, 4, and 5. The results showed that decreasing the swelling ratio will result in a more robust material since the G’ was 78.01 Pa for a swelling ratio of 20 and 2452.15 Pa for a swelling ratio of 5. Figure 4 illustrates pictures of sample 3 at different times at 130°C. After the brine was absorbed, the sample was placed in a high-temperature oven at 130°C, and pictures were taken every 30 minutes for visualization purposes. From these pictures, the particles of the HT-RPPG started to reassociate from each other after approximately an hour at 130°C. The particles gain more strength as they stay longer in the oven. Figure 5 illustrates an image for sample 3. The SEM image shows the pore structure for sample 3 after fully recrosslinked. The pores were strongly interconnected and bonded with the thick walls in between.

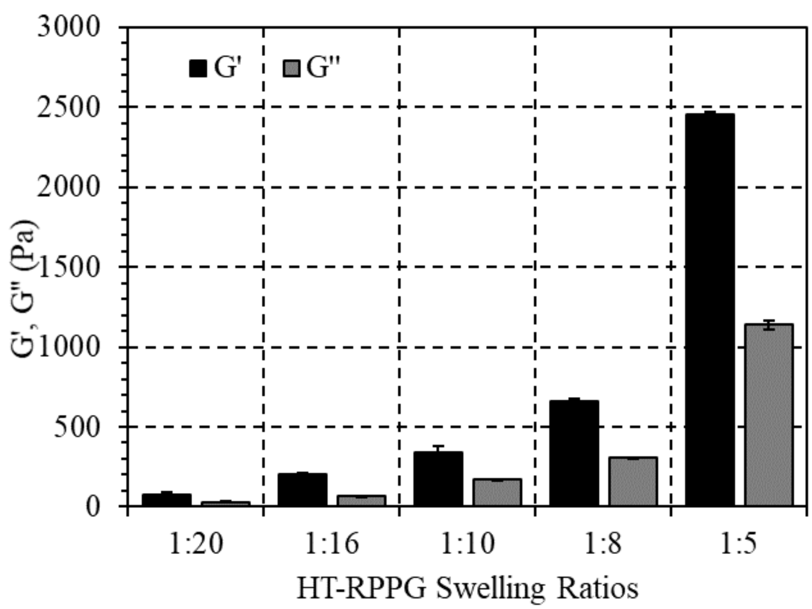

Figure 3. The G’ and G” for different samples

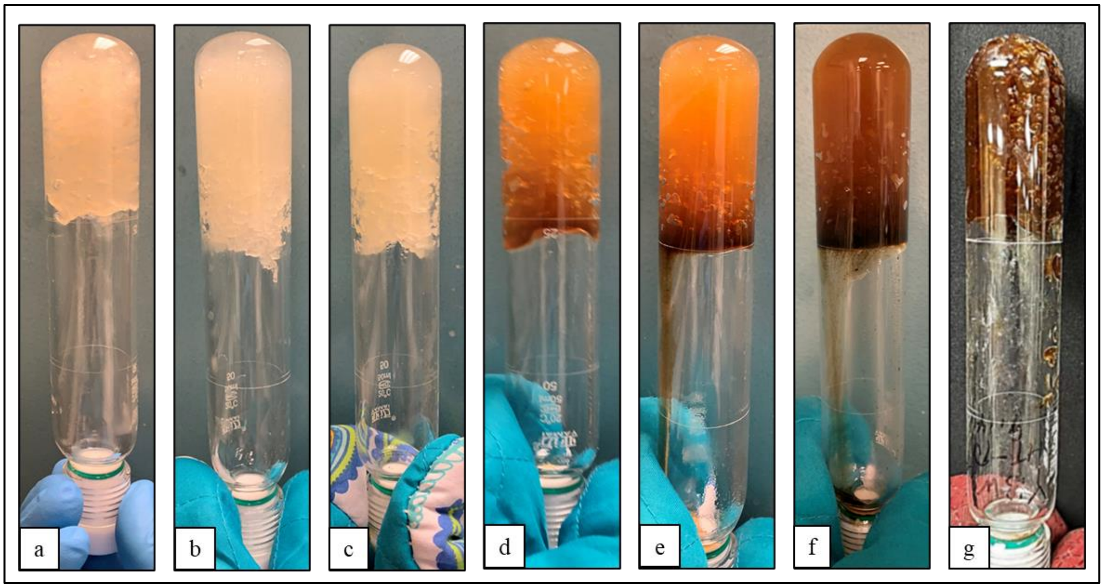

Figure 4. Pictures of sample 3 after aging for different times at 130 °C. (a) after absorbed all the brine. (b) after 30 minutes. (c) after 1 hour. (d) after 6 hours. (e) after 1 day. (f) after 3 days. (g) after 1 month.

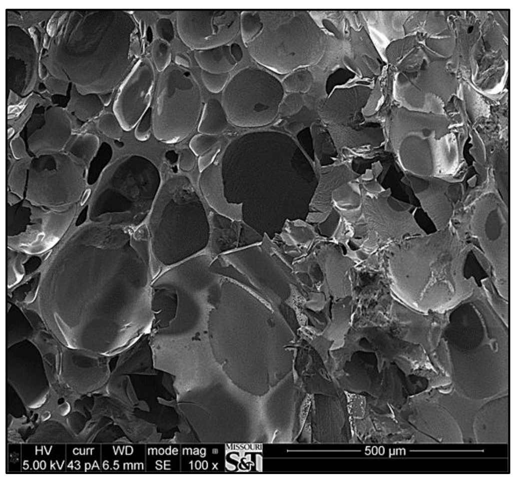

Figure 5. SEM image for sample 3 after fully recrosslinked.

5.2. CORE FLOODING EXPERIMENTS RESULTS

The evaluation of the plugging efficiency for the HT-RPPG was conducted utilizing a series of experiments using different samples and different fracture widths. In this section, the explanation of how the sealing pressures were measured will be discussed.

Figure 6 shows the core flooding test results for sample 3 utilizing a fracture width of 2.0 mm. As the graph illustrates, the HT-RPPG injection was stabilized at around 350 psi/ft after approximately 60 minutes. The entire HT-RPPG injection lasted about 80 minutes. The core was placed at 130°C for 24 hours. Then, the drilling fluid was injected using a flow rate of 1.0 cc/min to obtain the sealing pressure, which was 126.66 psi/ft.

Figure 7 shows the fractured cement core after the experiment. These pictures illustrate that even after the breakthrough has occurred, the gel is still inside the fracture, which reduced the volume of the fracture, leading to a significant reduction in permeability. Figure 8 shows the inside part of the fracture after the experiment. The fractured cement core was broken into two identical pieces to observe the surface of the fracture after the experiment. The HT-RPPG has clearly remained with a significant amount within the fracture which contributed enormously to decreasing the permeability within the fracture. Based on the obtained results, the permeability reduction was more than 99.99%. The permeability was decreased from 3.4 E+08 md to 6.82 md after treatment. Figure 9 shows the Frr results for sample 3 at different flow rates.

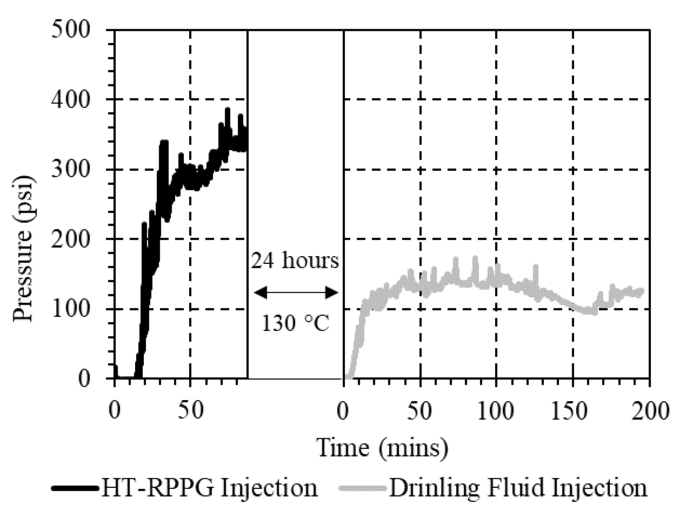

Figure 6. HT-RPPG core flooding results

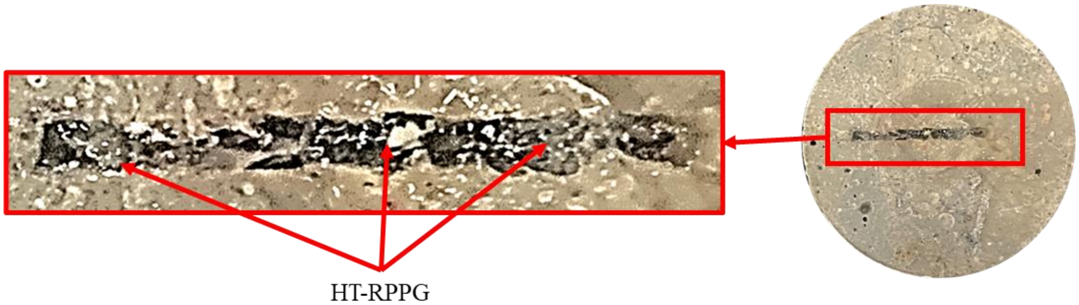

Figure 7. Fractured cement after the experiment

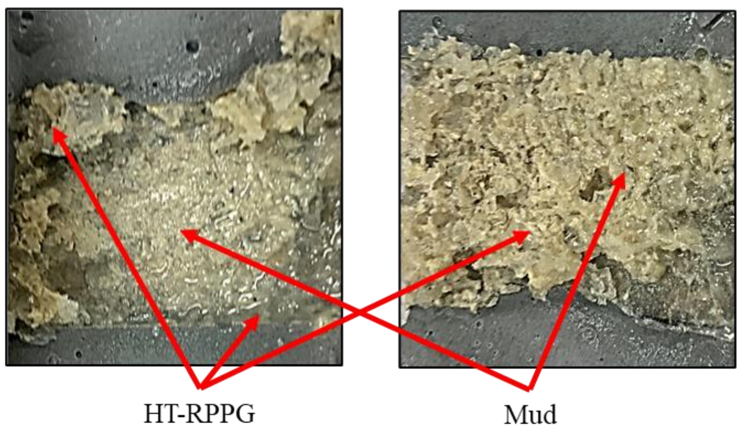

Figure 8. Inside image of the fracture for the fractured cement core after the experiment

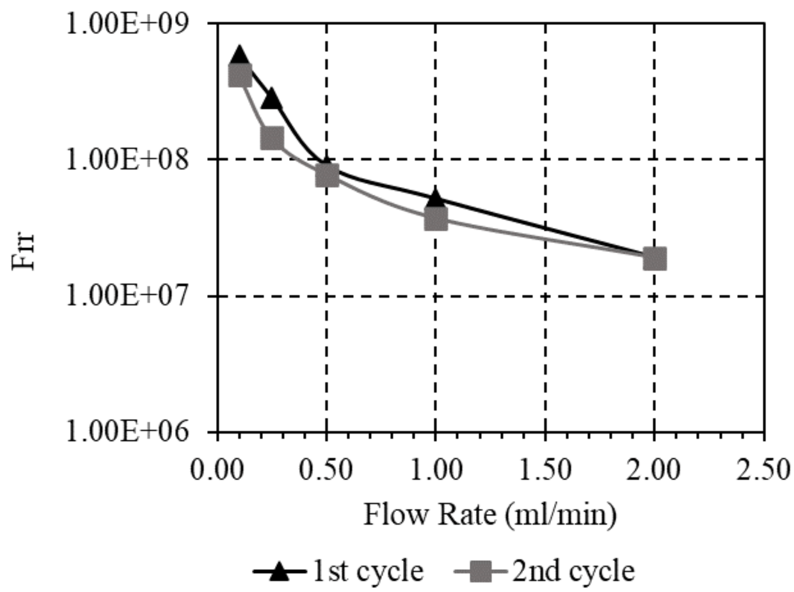

Figure 9. Frr results for sample 3.

5.2.1. Fracture Width Effect. As it is known, the lost circulation mainly occurs in fractured formations; while drilling through these formations, most or all of the drilling fluid will invade these fractures and result in severe or total loss. The fracture sizes play an essential role in this process; thus, investigating the HT-RPPG plugging efficiency at different fractures widths is a must as the fracture width and size in unknown in the real situations so it is necessary to have a robust product that can handle different fracture. Three different fracture widths were prepared for this study, including 1.50, 2.00, and 3.00 mm. The formulation used in these experiments was composed of brine and HT-RPPG with a swelling ratio of 10. Figure 10 shows sealing pressure results for different fracture widths. As expected, the results indicated that minuscule fractures would have the highest sealing pressure, but the exciting part is that the sealing pressure increased more than eight times when a fracture width of 1.50 was used compared to 2.00 mm. This indicates that the HT-RPPG is more likely to permanently close minor fractures. On the other hand, the sealing pressure was 88.29 psi/ft when a fracture width of 3.00 mm was used. Figure 11 shows the Frr results for different fracture widths at different flow rates. The permeability results showed a reduction of more than 99.99% after the treatment. Bai et al. (2022) claimed that their gel withstood a pressure of 623.3 psi/ft when a fracture of 1.00 mm was used at 160°C while our HT-RPPG could withstand the pressure of 1077 psi/ft when a fracture width of 1.50 mm was used.

5.2.2. Swelling Ratio Effect. The strength of the HT-RPPG mainly depends on its concentrations within the brine. Adding more HT-RPPG particles into the solution will undoubtedly increase its strength. However, this could affect the injection processes. Different ratios were prepared to evaluate the sealing pressure, including 1:5, 1:8, 1:10,

and 1:16. The experiments were conducted using a 2.00 mm fracture, and the waiting time was 24 hours at 130°C. For HT-RPPG swelling ratio of 5, the sample could not be injected into the fracture since after its particles absorbed the brine, they aggregated, as shown in Figure 12. The particles did not recrosslink but bonded, which prevented them from entering the fracture. However, this issue could be avoided when smaller particles is used.

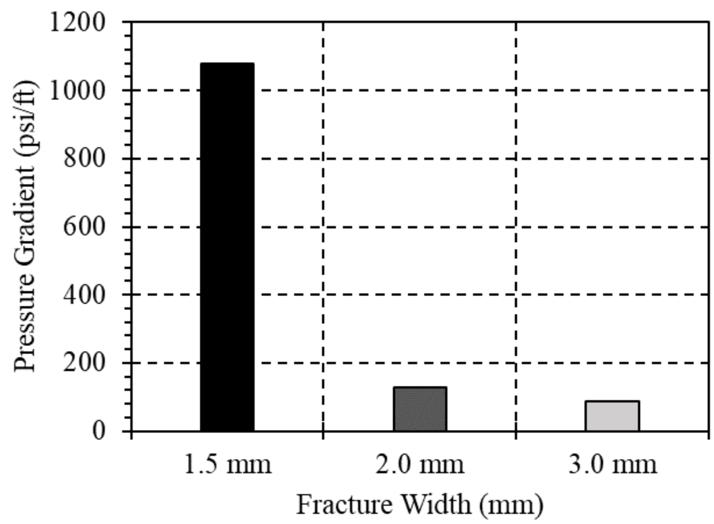

Figure 10. Sealing pressure at different fracture widths

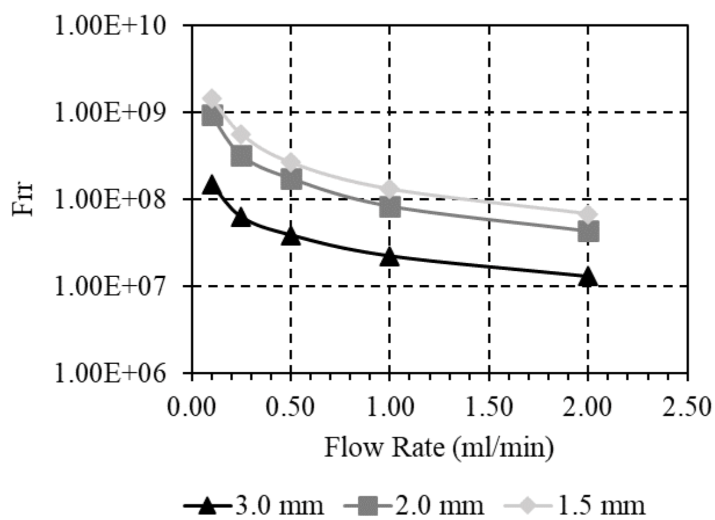

Figure 11. Frr results for different fracture widths

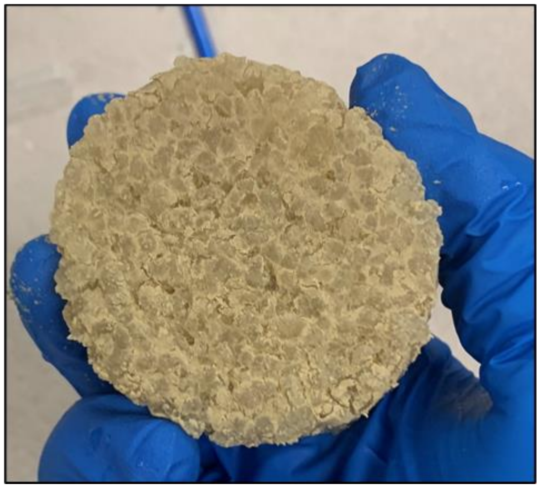

Figure 12. HT-RPPG sample after injection failure

The obtained results illustrate that reducing swelling ratios will facilitate gaining higher sealing pressure. Figure 13 shows the results of sealing pressure for 1:8, 1:10, and 1:16 swelling ratios. The sealing pressure was 44.42 psi/ft for the 1:16 swelling ratio and increased to 142.63 when the 1:8 swelling ratio was used. This coincides with what we mentioned earlier, that increasing the concentration of the HT-RPPG forms a more durable material that can withstand higher pressure before it cracks. The permeability results showed an enormous decrease as it became 6.59, 6.82, and 9.38 md for swelling ratios 8, 10, and 16, respectively, after treatment. Figure 14 shows the Frr results for each swelling ratio at different flow rates.

5.2.3. Drilling Fluid Effect. The effect of adding drilling fluid to the mixture on the material strength was evaluated. Four samples were prepared including two samples with swelling ratios of 20 and two samples with swelling ratios of 10 in the presence and absence of drilling fluid. Figure 15 shows the G' results for all the samples. As illustrated by the figure, the G' significantly increased drilling fluid in both swelling ratios when it

was introduced as it increased from 78.01 to 478.83 Pa for swelling ratios of 20 and from 341.87 to 944.38 Pa for swelling ratios of 10. This indicates that bentonite enhanced the strength of the material and formed a more robust material.

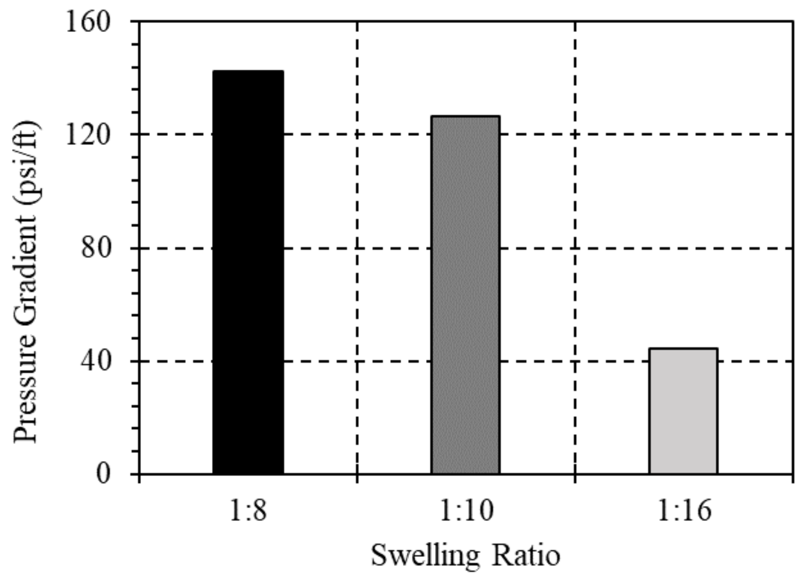

Figure 13. Sealing pressure at different swelling ratios

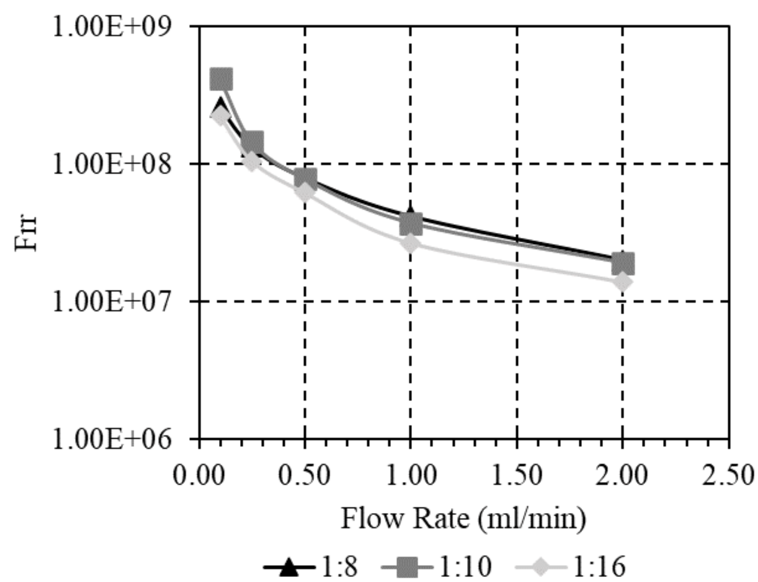

Figure 14. Frr results for different HT-RPPG swelling ratios

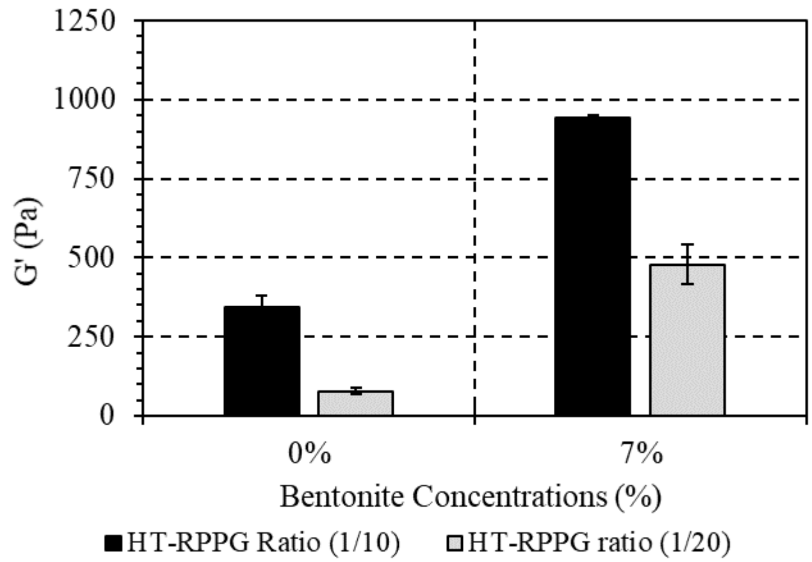

Figure 15. The G' results of HT-RPPG in presence and absence of drilling fluid

Moreover, core flooding experiments were conducted to investigate the impact of drilling fluid on the plugging efficiency of the HT-RPPG. The first sample was composed of brine and HT-RPPG with a swelling ratio of 10. The second sample was prepared by adding the HT-RPPG particles with a swelling ratio of 10 to the drilling fluid, which was prepared using 7% bentonite. The experiments were performed utilizing a 3.00 mm fracture width and 24 hours' waiting time at 130°C. Figure 16 shows the sealing pressure for both samples. The results showed that mixing the HT-RPPG with drilling fluid increased its ability to withstand higher pressure. The sealing pressure increased to 238.61 psi/ft when drilling fluid was introduced compared to 88.29 psi/ft when no bentonite was used. Figure 17 illustrates the Frr results for different bentonite concentrations using different flow rates. The permeability for each sample was reduced by more than 10^7 times as it became 25.56 and 18.33 md for 0 and 7% bentonite, respectively, after the treatment. Figure 18 shows the SEM image for the sample prepared using an HT-RPPG swelling ratio of 10 with

drilling fluid. As shown in the image, the drilling fluid did not affect the bonding between the pores as they still have a thick wall between them.

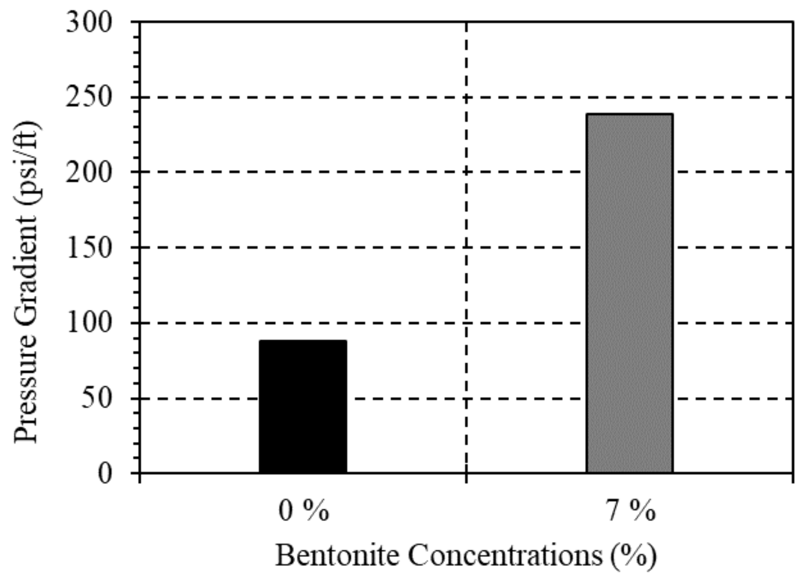

Figure 16. Sealing pressure results for different bentonite concentrations.

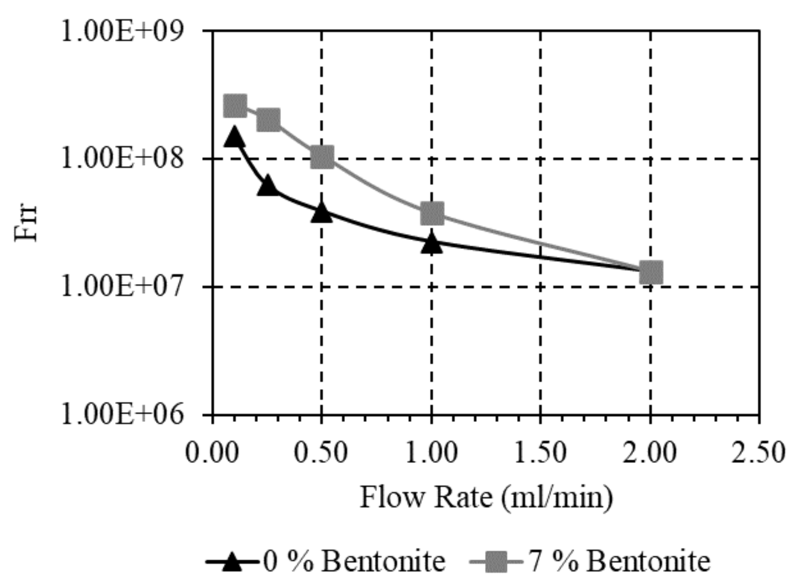

Figure 17. Frr results for different bentonite concentrations.

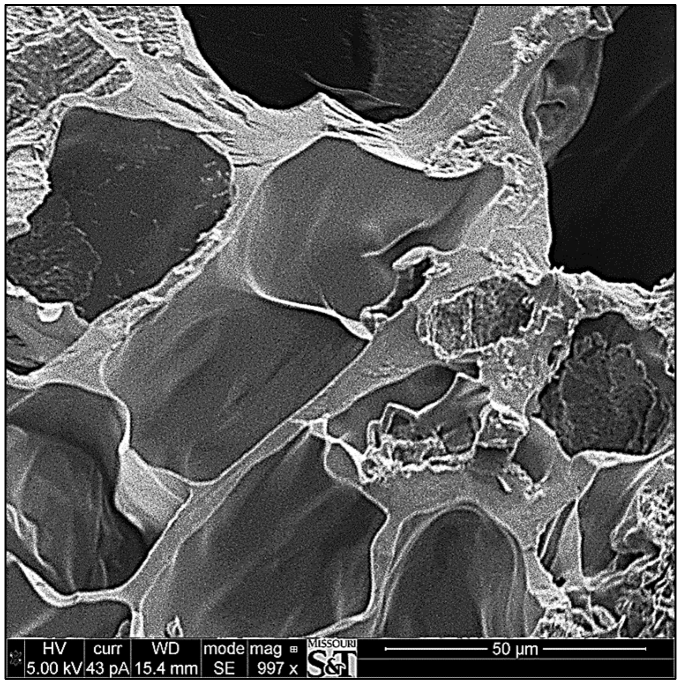

Figure 18. SEM image for HT-RPPG with swelling ratio of 10 mixed with drilling fluid

6. CONCLUSION

As high-temperature reservoirs have recently received attention, it is critical to have better materials available for the lost circulation application in those reservoirs. In this research, the capability of HT-RPPG to mitigate drilling fluid losses during drilling operations was investigated. It is approved that HT-RPPG has excellent plugging efficiency to fractures at 130°C. The HT-RPPG can be modified and used with different concentrations based on the fracture size and targeted formation.

The following conclusions were obtained from this research:

- Swelling ratios play a critical role in the material's strength. Reducing swelling ratios leads to a stronger material that can withstand a harsh environment. However,

this may lead to difficulty with injection capability which can be overcome by using smaller particles.

- HT-RPPG is a promising candidate to seal fractures at high-temperature reservoirs, 130°C, since it can hold up to 1077 psi/ft for a fracture width of 1.50 mm.
- Introducing drilling fluid to the mixture created a more robust material as it doubled its capability to withstand the pressure, as the sealing pressure increased from 88.29 to 238.61 psi when drilling fluid was introduced.
- The HT-RPPG remained in the fracture even after cracking, which significantly decreased the permeability, as the permeability decreased more than 10^7 times in all the experiments. This could play an essential role in reducing losses even if the pressure exceeds its capability.

ACKNOWLEDGEMENTS

The authors would like to thank the JIP members, including ConocoPhillips, Occidental Petroleum, and Daqing Wantong Chemical Plant, for their financial support.

NOMENCLATURE

HT-RPPG	High Temperature Re-Assembling Preformed Particle Gel
SEM	Scanning Electron Microscope
Frr	Residual Resistance Factor
SMP	Shape Memory Polymer
HMP Gel	Cross-linked Modified Polyacrylamide Gel

REFERENCES

Ahdaya, Mohamed, Al Brahim, Ali, Bai, Baojun, and Thomas Schuman. "Low-Temperature Recrosslinkable Preformed Particle Gel as a Material for Lost Circulation Control." SPE J. (2022;): doi: https://doi.org/10.2118/209783-PA

Ahdaya, M., & Imqam, A. (2019). Investigating geopolymer cement performance in presence of water based drilling fluid. Journal of Petroleum Science and Engineering, 176, 934-942. https://doi.org/10.1016/j.petrol.2019.02.010

Ab Lah, N. N., Ngah, K., & Sauki, A. (2019, November). Study on the viability of egg shell as a lost circulation material in synthetic based drilling fluid. In Journal of Physics: Conference Series (Vol. 1349, No. 1, p. 012135). IOP Publishing.

Alsaba, M., Nygaard, R., Hareland, G., & Contreras, O. (2014, April). Review of lost circulation materials and treatments with an updated classification. In AADE National Technical Conference and Exhibition, Houston, TX (pp. 1-9).

Bai, Y., Liu, C., Sun, J., Shang, X., Lv, K., Zhu, Y., & Wang, F. (2022). High Temperature Resistant Polymer Gel as Lost Circulation Material for Fractured Formation during Drilling. Colloids and Surfaces A: Physicochemical and Engineering Aspects, 128244.

Cook, J., Growcock, F., Guo, Q., Hodder, M., Van Oort, E., 2011. Stabilizing the wellbore to prevent lost circulation. Oilfield Rev. 23.

Cui, K., Jiang, G., Xie, C., Yang, L., He, Y., Shen, X., & Wang, X. (2021). A novel temperature-sensitive expandable lost circulation material based on shape memory epoxy foams to prevent losses in geothermal drilling. Geothermics, 95, 102145.

Deng, Z., Guancheng, J., Lili, Y., Yinbo, H., & Xiaoxiao, N. (2018). Microencapsulation of 2, 2′-Azobis (2-methylpropionamide) dihydrochloride initiator using acrylonitrile butadiene styrene as shell for application in lost-circulation control. Colloids and Surfaces A: Physicochemical and Engineering Aspects, 553, 134-142.

Du, G., Peng, Y., Pei, Y., Zhao, L., Wen, Z., & Hu, Z. (2017). Thermo-responsive temporary plugging agent based on multiple phase transition supramolecular gel. Energy & Fuels, 31(9), 9283-9289.

Ettehadi, A., & Altun, G. (2017). Extending thermal stability of calcium carbonate pills using sepiolite drilling fluid. Petroleum Exploration and Development, 44(3), 477-486.

Gamage, P., Deville, J. P., & Sherman, J. (2014). Solids-free fluid-loss pill for high-temperature reservoirs. SPE Drilling & Completion, 29(01), 125-130.

Growcock, F. B., Kaageson-Loe, N., Friedheim, J. E. A. 2., Sanders, M. W., & Bruton, J. (2009, March). Wellbore stability, stabilization and strengthening. In Offshore mediterranean conference and exhibition. OnePetro.

Hinkebein, T. E., Behr, V. L., & Wilde, S. L. (1983). Static slot testing of conventional lost circulation materials (No. SAND-82-1080). Sandia National Labs., Albuquerque, NM (USA).

Jiang, G., Deng, Z., He, Y., Li, Z., & Ni, X. (2019). Cross-linked polyacrylamide gel as loss circulation materials for combating lost circulation in high temperature well drilling operation. Journal of Petroleum Science and Engineering, 181, 106250. https://doi.org/-10.1016/j.petrol.2019.106250

Kulkarni, Sandeep D., Savari, Sharath , Gupta, Nivika , and Don Whitfill. "Designing Lost Circulation Material LCM Pills for High Temperature Applications." Paper presented at the SPE Deepwater Drilling and Completions Conference, Galveston, Texas, USA, September 2016. doi: https://doi.org/10.2118/180309-MS

Kumar, A., and Savari, S. 2011. Lost Circulation Control and Wellbore Strengthening: Looking Beyond Particle Size Distribution. AADE-11 NTCE-21, AADE National Technical Conference and Exhibition, Houston, Texas, USA, 12-14 April.

Kumar, A., Savari, S., Whitfill, D., Jamison, D.E., 2010. Wellbore strengthening: the less studied properties of lost-circulation materials. In: SPE Annual Technical Conference and Exhibition, Florence, Italy, 19-22 September.

Lecolier, Eric, Herzhaft, Benjamin, Rousseau, Lionel, Neau, Laurent, Quillien, Bernard, and Jacques Kieffer. "Development of a Nanocomposite Gel for Lost Circulation Treatment." Paper presented at the SPE European Formation Damage Conference, Sheveningen, The Netherlands, May 2005. doi: https://doi.org/10.2118/94686-MS

Mansour, A. K., Taleghani, A. D., & Li, G. (2017, June). Smart lost circulation materials for wellbore strengthening. In 51st US Rock Mechanics/Geomechanics Symposium. OnePetro.

Moore, P.L.: Drilling Practices Manual, 1986. Second Edition. PennWell Publishing Company

Sanders MW, Scorsone JT and Friedheim JE, (2010): High-fluid-loss, high-strength lostcirculation treatments. SPE Deepwater Drilling and Completions Conf., Galveston, Texas, USA;5-6 October, SPE Paper#135472

Sonmez, A., Kok, M. V., Bal, B., Bagatir, G., & Gucuyener, I. H. (2021). Comprehensive approach to torque and lost circulation problems in geothermal wells in terms of drilling fluid. Geothermics, 95, 102126.

Van Oort, E., Friedheim, J., Pierce, T., & Lee, J. (2011). Avoiding losses in depleted and weak zones by constantly strengthening wellbores. SPE Drilling & Completion, 26(04), 519-530.

Wang, B., Sun, J., Lv, K., Shen, F., & Bai, Y. (2021). Effects of a crosslinking agent on a supramolecular gel to control lost circulation. New Journal of Chemistry, 45(16), 7089-7095.

Wang, Bo., Sun, J., Shen, F., Lv, K., & Wang, J. (2021). Research on the rheological and flow characteristics of a supramolecular gel in fractures. Journal of Applied Polymer Science, 138(33), 50823.

White, Robert J. "Lost-circulation Materials and their Evaluation." Paper presented at the Drilling and Production Practice, New York, New York, January 1956.

Xie, B., Ma, J., Wang, Y., Tchameni, A. P., Luo, M., & Wen, J. (2021). Enhanced hydrophobically modified polyacrylamide gel for lost circulation treatment in high temperature drilling. Journal of Molecular Liquids, 325, 115155.

Yu, B., Zhao, S., Long, Y., Bai, B., & Schuman, T. (2022). Comprehensive evaluation of a high-temperature resistant re-crosslinkable preformed particle gel for water management. Fuel, 309, 122086.

SECTION

2. CONCLUSIONS AND RECOMMENDATIONS

2.1. CONCLUSIONS

This research comprehensively evaluated the capability of recrosslinkable materials to control severe losses during drilling applications under different temperature conditions (80 to 130 °C). Various methods were used to assess the possibility of RPPGs working as fluid loss agents. The plugging performance was studied using core flooding experiments, expressed by sealing pressure and residual resistance factor (Frr). Different factors were considered in order to better understand the performance of RPPGs under different circumstances, including the effect of swelling ratios, waiting time, fracture widths, mica concentrations, mica particle size distribution, walnut shell concentrations, and fiber concentration, as well as drilling fluid impact. A detailed summary of this research is as follows:

- The LT-RPPG which can recrosslink to form a strong bulk gel after being placed in fracture showed promising results to control fluid loss during drilling.
- The fracture width is inversely proportional to sealing pressure, as the sealing pressure reached 1074 psi/ft when the fracture width was 3.0 mm and 1381.5 psi/ft when the fracture width was 2.00 mm when a swelling ratio of 5 was used.
- The LT-RPPG swelling ratio is an important factor. Reducing swelling ratios will result in more robust material, which leads to higher sealing pressure as the sealing

pressure reached 1381 psi/ft when a swelling ratio of 5 was used for the fracture with a width of 2.0 mm.

- Waiting time plays an essential role in the sealing efficiency, as the LT-RPPG requires sufficient time to fully recrosslink to gain higher strength, which reflects on its ability to withstand higher pressure. However, the recrosslinking time is adjustable when a specific case is available.
- For all of the experiments, the fracture permeabilities were reduced more than 10^6 times after the treatment, indicating that the LT-RPPG has the potential of enhancing the plugging efficiency.
- Using drilling fluids instead of brine to prepare LT-RPPG can increase recrosslinked bulk gel strength, its sealing pressure as well as plugging efficiency to open fractures.
- Adding mica to the mixture of LT-RPPG and drilling mud resulted in more robust plugging materials; however, increasing the mica concentration from 1.00 to 2.00% did not significantly affect the gel strength but greatly increased the sealing pressure from 222.24 psi/ft to 357.75 psi/ft.
- The sealing pressure increased slightly when coarse mica was used compared to fine mica as it was 222.24 psi/ft when course mica was used compared to 182.21 psi/ft when fine mica was used.
- Walnut shell improved the sealing pressure significantly, especially when 1.00% was added as it reached 5685 psi/ft and no breakthrough was occurred.

- A strong MT-RPPG mixture can be achieved when using higher particle concentrations, which provided higher strength materials with G' as high as 5138.11 Pa for a swelling ratio of eight.
- Based on the results, the fracture width would not be an issue since the MT-RPPG can plug fracture widths from 1.5 mm up to 3.0 mm when using the proper swelling ratio.
- Waiting time has significant impact on sealing pressure as it increased from 197.77 to 718.44 psi/ft when waiting time was increased from 30 to 60 hours.
- Mixing the MT-RPPG with drilling fluid increased its strength significantly; however, it did not affect its ability to plug fractures.
- The remaining gel inside the fracture provided a high reduction in the permeability (more than 106 times), which indicates that even after the MT-RPPG reached its ability to withstand pressure, the reduction of the permeability significantly reduced the fluid loss.
- HT-RPPG is a promising candidate to seal fractures at high-temperature reservoirs, 130°C, since it can hold up to 1077 psi/ft for a fracture width of 1.50 mm.
- Introducing drilling fluid to the mixture created a more robust material as it doubled its capability to withstand the pressure, as the sealing pressure increased from 88.29 to 238.61 psi when drilling fluid was introduced.
- The HT-RPPG remained in the fracture even after cracking, which significantly decreased the permeability, as the permeability decreased more than 10^7 times in all the experiments. This could play an essential role in reducing losses even if the pressure exceeds its capability.

2.2. RECOMMENDATIONS

In this research, a comprehensive study was conducted to examine the capability of RPPG in controlling severe loss during drilling operations. Different RPPG products were evaluated under low, medium, and high temperature reservoir conditions. Several effects were considered in this study including the effect of the swelling ratios, the drilling fluid impact, the mica, walnut shell, and fiber effects, as well as waiting time influences and fracture widths impacts. The following recommendation should be considered for future work:

- Based on the loss mechanism, fluid loss is mainly encountered through different fractured formations such as carbonite formations; investigating actual fractured cores is recommended to understand the impact of matrix on the RPPGs' plugging performance under different circumstances.

- Since the fractures in actual formations are not ideal and form in cone shapes, examining ununiform fractures such as tapered fractures is a necessary step to investigate the capability of the RPPGs to control fluid losses under this phenomenon.

- Oil-based mud has been used widely during drilling operations, especially in high-pressure and high-temperature environments. Studying the effect of oil-based mud on the RPPG properties, such as strength, recrosslink time, swelling ratios, and plugging performance, is a must to better understand the behavior of the RPPGs under this situation, especially at high-pressure, high-temperature conditions.

- Investigating the ability of different RPPGs to seal fractured formations under high-pressure performance should be considered by using a back pressure regulator to understand the impact of varying pressure conditions on the plugging performance of the RPPGs.

- Since the RPPGs' particle size plays a critical role in the materials' injectivity and pumpability, studying different particle sizes is essential to understand their effect on the RPPGs performance and properties such as the recrosslinking time, swelling ratios, and strength.

- The bentonite can be impacted due to different conditions such as salinity and high temperature. Considering using different types of bentonites to examine its influence on the RPPGs’ performance is essential.

- In our research, we used the basic formula of water-based drilling fluid that contains water and 7.00% bentonite. It is crucial to study the effect of using water-based mud with its essential additives such as barite to understand the performance of RPPG in this situation.

BIBLIOGRAPHY

Ahdaya, M., & Imqam, A. (2019). Investigating geopolymer cement performance in presence of water based drilling fluid. Journal of Petroleum Science and Engineering, 176, 934-942.

Ahdaya, M., Al Brahim, A., Bai, B., & Schuman, T. (2022). Low-Temperature Recrosslinkable Preformed Particle Gel as a Material for Lost Circulation Control. SPE Journal, 1-11.

Al Maskary, S., Halim, A. A., & Al Menhali, S. (2014, November). Curing losses while drilling & cementing. In Abu Dhabi International Petroleum Exhibition and Conference. OnePetro.

Al-Arfaj, Mohammed , Amanullah, Md.. , and Raed Al-Ouhali. "Loss Circulation Materials Testing Methods: Literature Review." Paper presented at the SPE International Heavy Oil Conference and Exhibition, Kuwait City, Kuwait, December 2018. doi: https://doi.org/10.2118/193678-MS

Almagro, S. P. B., Frates, C., Garand, J., & Meyer, A. (2014). Sealing fractures: Advances in lost circulation control treatments. Oilfield Review, 26(3), 4-13.

Alsaba, M., Nygaard, R., Hareland, G., & Contreras, O. (2014, April). Review of lost circulation materials and treatments with an updated classification. In AADE National Technical Conference and Exhibition, Houston, TX (pp. 1-9).

Alsaba, Mortadha Turki, "Investigation of lost circulation materials impact on fracture gradient" (2015). Doctoral Dissertations. 2437.

Benyeogor, O., Awe, S., Amah, O., Ugochukwu, O., Erinle, A., Akinfolarin, A., & Oseme, U. (2016, December). Wellbore Strengthening in Narrow Margin Drilling. In SPE/AAPG Africa Energy and Technology Conference. Society of Petroleum Engineers.

Canson, B. E. 1985. Lost Circulation Treatment for Naturally Fractured, Vugular, or Cavernous Formations. SPE/IADC 13440, SPE/IADC Drilling Conference, New Orleans, USA, 5-8 March.

Cook, J., Growcock, F., Guo, Q., Hodder, M., & van Oort, E. (2011). Stabilizing the wellbore to prevent lost circulation. Oilfield Review, 23(4), 26-35.

Du, G., Peng, Y., Pei, Y., Zhao, L., Wen, Z., & Hu, Z. (2017). Thermo-responsive temporary plugging agent based on multiple phase transition supramolecular gel. Energy & Fuels, 31(9), 9283-9289.

Feng, Y., Jones, J. F., & Gray, K. E. (2016). A review on fracture-initiation and-propagation pressures for lost circulation and wellbore strengthening. SPE Drilling & Completion, 31(02), 134-144.

Fitch, J. P., & Minter, R. B. (1976, January). Chemical diversion of heat will improve thermal oil recovery. In SPE Annual Fall Technical Conference and Exhibition. Society of Petroleum Engineers.

Hettema, M. H., Taugbol, P., Friedheim, K., Huynh, J., & Sanders, H. MW and Young, S. 2007."Development of an Innovative High-Pressure Testing Device for the Evaluation of Drilling Fluid Systems and Drilling Fluid Additives within Fractured Permeable Zones" (Vol. 82). 0MC-2007.

Howard, G. C., & Scott, P. P. (1951). An analysis and the control of lost circulation. Journal of Petroleum Technology, 3(06), 171-182.

Jeennakorn, Montri, "The effect of testing conditions on lost circulation materials' performance in simulated fractures" (2017). Doctoral Dissertations. 2599.

Kefi, S., Lee, J. C., Shindgikar, N. D., Brunet-Cambus, C., Vidick, B., & Diaz, N. I. (2010, November). Optimizing in four steps composite lost-circulation pills without knowing loss zone width. In IADC/SPE Asia Pacific Drilling Technology Conference and Exhibition. OnePetro.

Kumar, A., and Savari, S. 2011. Lost Circulation Control and Wellbore Strengthening: Looking Beyond Particle Size Distribution. AADE-11 NTCE-21, AADE National Technical Conference and Exhibition, Houston, Texas, USA, 12-14 April.

Kumar, A., Savari, S., Jamison, D. E., & Whitfill, D. L. (2011, April). Lost circulation control and wellbore strengthening: looking beyond particle size distribution. In AADE national technical conference and exhibition, Houston, Texas, USA (pp. 12-14).

Kumar, A., Savari, S., Whitfill, D. L. et al. . 2010. Wellbore Strengthening: The Less-Studied Properties of Lost-Circulation Materials. Paper presented at theSPE Annual Technical Conference and Exhibition, Florence, Italy, 19–22 September. SPE-133484-MS. 10.2118/133484-MS.

Long, Y., Bai, B., & Schuman, T. P. (2020). Re-Crosslinking Particle Gel for Co_2 Conformance Control and CO_2 Leakage Blocking.

Moore, P. L. (1986). Drilling practices manual.

Nayberg, T. M., & Petty, B. R. (1986, April). Laboratory study of lost circulation materials for use in oil-base drilling muds. In SPE Deep Drilling and Production Symposium. OnePetro.

Pu, J., Bai, B., Alhuraishawy, A., Schuman, T., Chen, Y., & Sun, X. (2019). A recrosslinkable preformed particle gel for conformance control in heterogeneous reservoirs containing linear-flow features. SPE Journal, 24(04), 1714-1725.

Salehi, S., & Nygaard, R. (2012, October). Numerical modeling of induced fracture propagation: a novel approach for lost circulation materials (LCM) design in borehole strengthening applications of deep offshore drilling. In SPE annual technical conference and exhibition. OnePetro.

Sanders, M. W., Scorsone, J. T., & Friedheim, J. E. (2010, October). High-fluid-loss, high-strength lost circulation treatments. In SPE deepwater drilling and completions conference. OnePetro.

Sanders, M. W., Young, S., & Friedheim, J. (2008, April). Development and testing of novel additives for improved wellbore stability and reduced losses. In AADE fluids conference and exhibition, Houston, USA (pp. 8-9).

Song, T., Ahdaya, M., Zhao, S., Zhao, Y., Schuman, T., & Bai, B. (2022). Evaluation of a Novel Recrosslinkable Hyperbranched Preformed Particle Gel for the Conformance Control of High-Temperature Reservoirs with Fractures. SPE Journal, 1-13.

Song, T., Zhai, Z., Liu, J., Eriyagama, Y., Ahdaya, M., Alotibi, A., ... & Bai, B. (2022). Laboratory evaluation of a novel Self-healable polymer gel for CO2 leakage remediation during CO2 storage and CO2 flooding. Chemical Engineering Journal, 444, 136635.

Suyan, K. M., Dasgupta, D., Sanyal, D., Sharma, V., & Jain, V. K. (2007, November). Managing total circulation losses with crossflow while drilling: case history of practical solutions. In SPE Annual Technical Conference and Exhibition. OnePetro.

Van Oort, E., Friedheim, J., Pierce, T., & Lee, J. (2011). Avoiding losses in depleted and weak zones by constantly strengthening wellbores. SPE Drilling & Completion, 26(04), 519-530.

White, Robert J. "Lost-circulation Materials and their Evaluation." Paper presented at the Drilling and Production Practice, New York, New York, January 1956.

Whitfill, D., & Miller, M. (2008, April). Developing and testing lost circulation materials. In AADE fluids conference and exhibition, Houston, Texas, USA (pp. 8-9).

VITA

Mohamed Saad Mohamed Ahdaya was born in Tripoli, Libya. He received his bachelor’s degree in petroleum engineering from University of Tripoli, Tripoli, Libya, in 2012. He started at Missouri University of Science and Technology during the summer semester of 2016. He earned the best graduate student research poster among the Geoscience and Geology and Petroleum Engineering award in 2018. He received his Master of Science degree in Petroleum Engineering from Missouri University of Science and Technology in December 2018. He received his PhD in Petroleum Engineering from Missouri University of Science and Technology in July 2022.