
Masters Theses

Student Theses and Dissertations

Fall 2016

An investigation of plugging and increasing oil recovery in sandstone porous media by micrometer-size particle gel

Haifeng Ding

Follow this and additional works at: https://scholarsmine.mst.edu/masters_theses

 Part of the [Petroleum Engineering Commons](#)

Department:

Recommended Citation

Ding, Haifeng, "An investigation of plugging and increasing oil recovery in sandstone porous media by micrometer-size particle gel" (2016). *Masters Theses*. 7595.

https://scholarsmine.mst.edu/masters_theses/7595

This thesis is brought to you by Scholars' Mine, a service of the Missouri S&T Library and Learning Resources. This work is protected by U. S. Copyright Law. Unauthorized use including reproduction for redistribution requires the permission of the copyright holder. For more information, please contact scholarsmine@mst.edu.

AN INVESTIGATION OF
PLUGGING AND INCREASING OIL RECOVERY
IN SANDSTONE POROUS MEDIA
BY MICROMETER-SIZE PARTICLE GEL

by

HAIFENG DING

A THESIS

Presented to the Faculty of the Graduate School of the
MISSOURI UNIVERSITY OF SCIENCE AND TECHNOLOGY

In Partial Fulfillment of the Requirements for the Degree

MASTER OF SCIENCE

in

PETROLEUM ENGINEERING

2016

Approved
Dr. Baojun Bai, Advisor
Dr. Mingzhen Wei
Dr. Peyman Heidari

© 2016

Haifeng Ding

All Rights Reserved

ABSTRACT

Micrometer-size particle gel (microgel) has been developed and successfully applied to improve conformance in mature reservoirs. However, quite few researches have been published to address on their effect on oil recovery and rock permeability modification.

This work conducted a series of core flooding experiments to investigate the injectivity, plugging efficiency and improving oil recovery potentials of a couple of microgels synthesized in our lab.

First, this work studied the impact of permeability and crosslinker concentration on PAM type microgel treatment. Results shows that microgel treatment would have a less plugging efficiency in lower permeability rocks, which could be caused by high injection pressure in lower permeability rocks. However, the oil recovery was higher in lower permeability rocks. The microgel synthesized by lower concentration of crosslinker have bigger particle sizes. Meantime, there would be higher plugging efficiency and better improved oil recovery when crosslinker concentration is lower.

Moreover, this work also studied the effect of permeability and oil saturation on the treatment of a novel type of microgel which can re-crosslink at specific reservoir conditions. Different from PAM microgel, the re-crosslinked microgel would have both better plugging efficiency and oil recovery improvement in lower permeability rocks. Meanwhile, microgel have better transportation ability in the rocks with high oil saturation than those without oil.

Comparing Re-crosslinked microgel to PAM microgel, Re-crosslinked microgel have a better strength and disproportionate permeability reduction effect.

ACKNOWLEDGMENTS

First, I would like to thank my advisor, Dr. Baojun Bai. His guidance, advices and support help me complete this work. His patience, erudition and diligence make me enjoy the time in this university.

I would like to thank my committee members, Dr. Mingzhen Wei and Dr. Peyman Heidari for their suggestion and insight.

I would also like to give my deeply appreciation to Dr. Abdulmohsin Imqam, Xindi Sun, Jiaming Geng and Ayman Almohsin for helping and teaching me almost everything in the lab. In addition, I want to thank all my colleagues in lab especially Jingyang Pu, Ze Wang, Sujay Suresh, Yifu Long and Ali Alhuraishawy for their help during running experiments.

My family especially my late maternal grandparents also show their incredible support and love to me. No word can describe my appreciation to them.

I also want to express my appreciation to Missouri University of Science and Technology and UM system for providing me a great research environment.

TABLE OF CONTENTS

	Page
ABSTRACT.....	iii
ACKNOWLEDGMENTS	iv
LIST OF ILLUSTRATIONS	viii
LIST OF TABLES	x
NOMENCLATURE	xi
SECTION	
1. INTRODUCTION.....	1
1.1. BACKGROUND.....	1
1.2. OBJECTIVES	1
1.3. SCOPE OF THIS RESEARCH.....	2
2. LITERATURE REVIEW.....	4
2.1. ENHANCED OIL RECOVERY AND CONFORMANCE CONTROL.....	4
2.1.1. Enhanced Oil Recovery (EOR).....	4
2.1.1.1 Thermal EOR methods.....	4
2.1.1.2 Miscible or solvent injection EOR methods.....	5
2.1.1.3 Chemical EOR methods.	5
2.1.2. Excessive Water Production and Reservoir Heterogeneity.	5
2.1.3. An Introduction of Gel Treatment.	6
2.1.3.1 In-situ gel system.....	6
2.1.3.2 Preformed gel system.	8
2.1.4. Disproportionate Permeability Reduction (DPR) of Gel Treatment.....	11

2.2. PREVIOUS LAB WORKS WITH NANO-SPHERE OR MICRO-SPHERE USING NON-FRACTURE MODEL.....	12
3. PAM MICROGEL	18
3.1. EXPERIMENTAL DESCRIPTION	18
3.1.1. Materials.	18
3.1.2. Experimental Setup.....	19
3.1.3. Experimental Procedures..	20
3.1.4. Experimental Plans.....	23
3.2. RESULTS FROM EXPERIMENTS	23
3.2.1. The Effect of Permeability on Microgel Treatment.	23
3.2.2. The Effect of Crosslinker Concentration on Microgel Treatment.	32
3.3. DATA ANALYSIS AND DISCUSSION	39
3.4. SUMMARY.....	42
4. RE-CROSSLINKED MICROGEL	43
4.1. EXPERIMENTAL DESCRIPTION	43
4.1.1. Materials.....	43
4.1.2. Experimental Plans.....	43
4.1.2.1 The plugging ability of Re-crosslinked microgel in single phase condition	43
4.1.2.2 The plugging and increasing oil recovery abilities of Re-crosslinked microgel in multiple-phase condition	46
4.1.2.3 The impact of oil phase and oil saturation on microgel transportation	46
4.2. RESULTS FROM EXPERIMENTS	47
4.2.1. Experiments with Single Phase.....	47

4.2.2. Experiments with Multiple Phases.....	48
4.2.3. The Effect of Oil Phase on Gel Injection Process.....	54
4.3. DATA ANALYSIS AND DISCUSSION.....	57
4.4. SUMMARY.....	58
5. CONCLUSION	59
6. FUTURE WORKS	61
BIBLIOGRAPHY	62
VITA.....	65

LIST OF ILLUSTRATIONS

	Page
Figure 1.1. Scope of this work	3
Figure 2.1. The mechanism of in-situ gel	7
Figure 2.2. Comparison of millimeter PPG before and after swelling	9
Figure 2.3. Mechanism of conformance control using Brightwater®	11
Figure 2.4. Core flooding experiments setup.....	14
Figure 2.5. Experiments setup of non-crossflow heterogeneous model	16
Figure 3.1. Schematic diagram of setup of experiments.....	19
Figure 3.2. Experimental flow chart	22
Figure 3.3. Gel injection pressure plots with different permeability	26
Figure 3.4. Second water flooding pressure plots with different permeability.....	27
Figure 3.5. Resistance factor and the peaks of gel injection and second water flooding .	27
Figure 3.6. Residual resistance factor and the peaks of second water flooding	28
Figure 3.7. Residual resistance factor to water	29
Figure 3.8. Residual resistance factor to oil.....	30
Figure 3.9. Oil recovery plot of experiment #1	30
Figure 3.10. Oil recovery plot of experiment #3	31
Figure 3.11. Oil recovery plot of experiment #6	31
Figure 3.12. Gel injection pressure plots with different crosslinker concentration	34
Figure 3.13. Second water flooding pressure plots with different crosslinker concentration.....	34
Figure 3.14. Resistance factor and the peaks of gel injection pressure	35

Figure 3.15. Residual resistance factor and the peaks of gel injection pressure.....	35
Figure 3.16. Residual resistance factor for water	36
Figure 3.17. Residual resistance factor for oil	36
Figure 3.18. Oil recovery plot of experiment #2	37
Figure 3.19. Oil recovery plot of experiment #4	37
Figure 3.20. Oil recovery plot of experiment #5	38
Figure 3.21. RRF at 1 ml/min for oil and water.....	41
Figure 4.1. Size distributions of Re-crosslinked microgel particles in different conditions.....	45
Figure 4.2. Experimental workflow	47
Figure 4.3. Difference among experiments by work flow	49
Figure 4.4. Gel injection pressure plots	51
Figure 4.5. Second water flooding pressure plots.....	51
Figure 4.6. Peaks of resistance factor during gel injection and the resistance factor	52
Figure 4.7. The peaks of residual resistance factor and the residual resistance factor	52
Figure 4.8. Residual resistance factors of water and oil in experiment #C	53
Figure 4.9. Residual resistance factors of water and oil in experiment #D	53
Figure 4.10. Residual resistance factors of water and oil in experiment #E.....	54
Figure 4.11. Gel injection pressure plot of experiment #G.....	55
Figure 4.12. Gel injection pressure plot of experiment #H.....	56
Figure 4.13. Gel injection pressure plot of experiment #E	56

LIST OF TABLES

	Page
Table 2.1. Oil recovery improvement using different models	17
Table 2.2. Injection profile improvement using different models	17
Table 3.1. Component and proportion of each PAM samples	18
Table 3.2. Injection velocity and their corresponding pump rates conversion table	21
Table 3.3. Crosslinker concentration and particle sizes after swelling	24
Table 3.4. Experiments performed in this study	24
Table 3.5. Pore sizes and particle size contrast	25
Table 3.6. Oil recovery factors in different stages	32
Table 3.7. Particle/pore size contrast ratio	33
Table 3.8. Oil recovery factors in different stages	38
Table 3.9. Injection pressure at 1ml/min in experiment #1, 3 and 6	40
Table 4.1. Core samples' permeability and contrast ratio to particle	44
Table 4.2. Injection flow rates and their corresponding velocities	44
Table 4.3. Injection flow rates and their corresponding velocities	48
Table 4.4. Core samples' permeability and contrast ratio to particle	49
Table 4.5. RRFs of experiment #A and #B with different flow rates	50
Table 4.6. Oil recovery factors of different stages	55

NOMENCLATURE

Symbol	Description
F_{rr}	Residual resistance factor
RRF	Residual resistance factor
F_r	Resistance factor
RF	Resistance factor
M	Mobility ratio
λ	Mobility
K	Permeability
μ	Viscosity
ΔP	Injection pressure
η	Plug efficiency
d	Pore size
Φ	Porosity

1. INTRODUCTION

1.1. BACKGROUND

Only one third of initial oil in place would be produced after primary and secondary recovery stages. With the current low oil price, drilling new wells is not always an economical method. As a result, enhanced oil recovery (EOR) process is necessary and cost efficient.

The heterogeneity of reservoir is always responsible for low oil recovery and excessive water production. Excessive water production will cause problems from both economic and environmental aspects. At this circumstance, conformance control is a necessary method to solve this problem.

Gel treatment has been proved as a cost-efficiency conformance control method. During secondary recovery stage like water flooding, injection fluids always have a trend to go through higher permeability zone, which would cause low sweep efficiency and high remaining oil saturation. Thus gel treatment is designed to plug higher permeability zone to increase sweep efficiency then obtain higher oil recovery and lower water production. To plug fractures in reservoirs, bigger size preformed particle gels are often applied to improve injection profile. To improve conformance in low permeability and non-fracture reservoir, smaller size particle gel like micrometer-size particle gel would be a suitable option.

1.2. OBJECTIVES

The primary objective of this research is to investigate the factors that have impacts on plugging and oil recovery improvement abilities of microgel. Core flooding

experiments will be performed with two types of microgels: PAM microgel and Re-crosslinked microgel. The following objectives were established for this research:

1. When studying PAM microgel, microgels were synthesized by different concentrations of crosslinker to study the effect of crosslinker concentration. Meanwhile, core samples with different permeability ranges were selected in core flooding experiments to study the impact of rock permeability.
2. For Re-crosslinked microgel, different permeability ranged core samples were used to determine the impact of rock permeability. At the same time, the effect of oil saturation on gel transportation and oil recovery was studied.
3. By performing experiments using two types of microgels, this work compared the properties of both microgels.

1.3. SCOPE OF THIS RESEARCH

This research was primarily a laboratory study to investigate the factors affecting microgels' plugging and oil recovery improvement abilities.

First, six core flooding experiments using PAM microgel were performed with different crosslinker concentrations and different permeability ranges. Those experiments could be divided into two groups: experiments with same rock permeability range and experiments using microgels with same crosslinker concentration.

Then, eight more core flooding experiments using Re-crosslinked microgel were performed. Four experiments containing residual oil and two experiments without residual oil were performed to study the impact of permeability. Meanwhile, two more

experiments were performed to study the oil saturation's effect on microgel particle transportation.

Figure 1.1 shows the scope of this work.

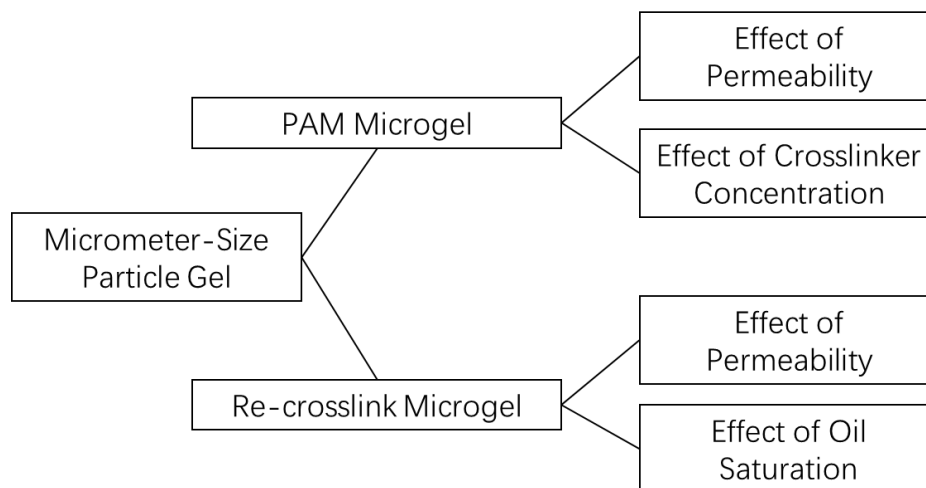


Figure 1.1. Scope of this work

2. LITERATURE REVIEW

2.1. ENHANCED OIL RECOVERY AND CONFORMANCE CONTROL

2.1.1. Enhanced Oil Recovery (EOR). There are three main oil recovery mechanisms: primary recovery, secondary recovery and enhanced oil recovery (EOR) which is also known as tertiary recovery. During primary recovery, oil is produced by nature energy of reservoirs. Such energy are solution-gas drive, gas-cap drive, fluid/rock expansion, gravity drainage and natural water drive [1]. After the initial energy of a reservoir has depleted, fluids such as water and gas would be injected into reservoir to replace oil as secondary recovery.

After primary and secondary recovery, about 30% of initial oil in place would be produced [2]. Then 70% of initial oil in place become a target of enhanced oil recovery. Normally, EOR methods are classified into three categories: thermal, miscible or solvent injection and chemical [3].

2.1.1.1 Thermal EOR methods. In heavy oil reservoir, thermal EOR methods normally applied to reduce oil viscosity. In light oil reservoir, thermal energy could vaporize oil into solvent front.

Huff and puff is one of the most common thermal methods. In this method, steam is injected into well for a period which normally is 2-4 weeks. Then the well would be shut in for days to let the formation “soaked”. The oil rate would be high due to the lower viscosities and higher reservoir pressure following the high temperature. Such process could be repeated after oil rate back to a predetermined level. The other thermal methods including steam flooding, combustion, hot water flooding, and so on.

2.1.1.2 Miscible or solvent injection EOR methods. Miscible or solvent injection methods can be used to increase recovery from the miscibility or displacement between oil and injected fluids such as hydrocarbon solvents, carbon dioxide and nitrogen [1]. There are two major situations in such methods: 1) First-contact-miscible. It is the more effective situation. During this process, oil would be miscible and produced with injection fluid. 2) Multiple-contact-miscible. injection fluids would not be miscible with oil in a reservoir. The injection phase would displace oil to increase recovery. However, the injection phase could be miscible with oil phase with proper pressure, temperature and composition.

2.1.1.3 Chemical EOR methods. Chemical methods involve the injection of specific fluids into a reservoir to increase oil recovery by wettability alternation, interfacial tension reduction, mobility control or conformance control. Surfactant is often used to reduce the interfacial tension between oil and water and change the wetting phase of reservoir to make a favorable situation to produce more oil. Polymer flooding is a common method for mobility control since the viscosity of displacing phase would be increased when adding polymer. Gel treatment is a widely applied method for conformance control. By injecting gels, the permeability of higher permeability zones of formations could be reduced and thus more oil could be recovered from previously unswept lower permeability zones.

2.1.2. Excessive Water Production and Reservoir Heterogeneity. Excessive water production is a major challenge of oil industry in both economics and environments. Environmentally, produced water could do environment damage as a source of pollution. Economically, excessive water production will cause corrosion of

facilities. Besides, the cost of disposing of produced water is high: it cost 5 cents to more than 50 cents to handle every barrel of water. What is more, when water cut is higher than 80%, it cost 4 dollars per barrel of oil produced to handle water problem.

Worldwide, an average of 210 barrels of water will be produced along with every 75 barrels of oil [4]. Excessive water production could eventually cause shutting down even abandon of a well.

The heterogeneity of reservoirs is a major reason caused excessive water production, which could lead to poor sweep efficiency and watered-off layers. Conformance control is always concerned as an effective solution for heterogeneity problem of reservoir.

2.1.3. An Introduction of Gel Treatment. Gel treatment is a cost-effective chemical EOR method which is widely applied to improve injection profile as conformance control. During injection stage, injected fluids always have a trend to go through higher permeability zone, which would cause low sweep efficiency and high remaining oil saturation. Thus gel treatment is designed to plug higher permeability zone to increase sweep efficiency to obtain higher oil recovery and lower water production. Generally, there are two major gel treatment system: in-situ gel system and preformed gel system.

2.1.3.1 In-situ gel system. The gelant, which is often composed of polymer, crosslinker and additives, is injected into a reservoir before gelling, and the gelation occurs in reservoirs.

Figure 2.1 [5] shows how gelant is injected into a well and become gel after gelation. First, gelant is injected in to formation as shown in figure a. Then, water is

injected to push gelant into the formation as shown in b). Because of the permeability difference, more gelant is placed in higher permeability zones. Then the well is shut in to allow gelation occur as figure c). Then, water is re-injected as showed in figure d), more water would go through lower permeability zones and thus sweep efficiency is increased.

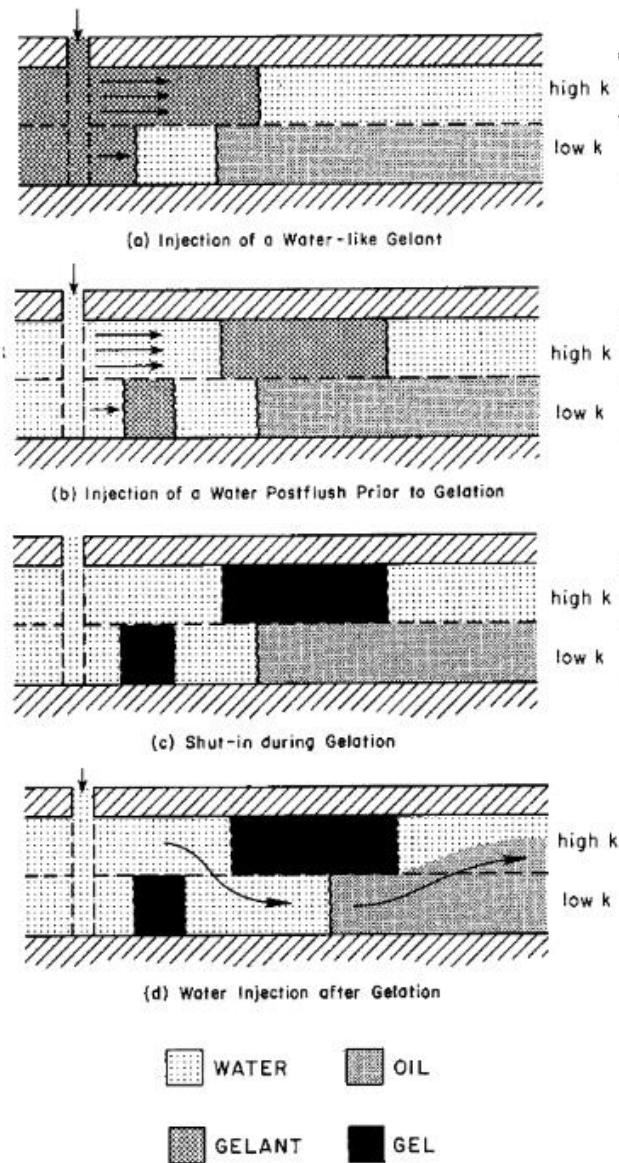


Figure 2.1. The mechanism of in-situ gel [5]

There are two categories of in-situ gels: monomer gels and polymer gels. In monomer gel system, water-like monomer solution is injected into the formation and then well is shut in for monomer to polymerize [6]. However, the difficulty of gelation control, environmental and health risk caused by monomer's toxic makes this treatment unpopular in the oil industry.

Polymer gels are widely applied in oil industry nowadays since such technology is economic viable [7] and relative environmentally friendly. These gels can be formed by synthetic polyacrylamides or polysaccharides. Since their better stability, polyacrylamides are more common. Normally, polymer gels are formed with particle hydrolyzed polyacrylamides, crosslinkers and some additives [8].

2.1.3.2 Preformed gel system. Using preformed gels to control conformance is a newer trend of gel treatment, which gel is already fully prepared before injection, also known as preformed particle gel (PPG). Different from in-situ gel, PPG is formed and crosslinked at surface facilities rather than in formation after injection. Therefore, the gelation can be controlled. Preformed particle gels could be classified into three types based on their particle sizes: millimeter-size preformed particle gel, micrometer-size preformed particle gel and nanometer-size preformed particle gel.

Millimeter-size preformed particle gel. Millimeter-size PPG is developed by Petro China and Missouri S&T. It is an improved super adsorbent polymer, also known as SAP. Such materials could absorb over hundred times as their weight in liquid and stay stable under high pressure [9]. Figure 2.2 [10] show the pictures of a PPG sample before and after swelling. The size of PPG usually ranges from 10 micrometers to a few centimeters, depending the features of target zones. Comparing to in-situ polymer gels,

millimeter PPG could resist higher temperature (up to 120 degrees centigrade) and any kind of brines. Besides, it is easy and quick to prepare since it can be mixed in any convenient water and can well be dispersed in short period. It is also easy to be monitored during injection process and environmental friendly.



Figure 2.2. Comparison of millimeter PPG before and after swelling [10]
Left tube: dried particles; right tube: swelling particles

In 1999, mm-size PPGs were first successfully applied in a high temperature and high salinity reservoir in China by SINOPEC [9]. Since then, such gel becomes one of the most widely applied conformance control technologies. By 2015, mm-size PPGs have been applied in nearly 10,000 wells in China [10]. However, millimeter-size PPGs can only be used to plug high permeability channels or fractures because of their large size.

However, PPGs with smaller particle sizes are required when dealing with lower permeability reservoirs.

Micrometer-size preformed particle gel. Institut Français du Pétrole (IFP) reported a type of microgels with the size ranging from 0.1 to 10 micrometers in 2001 [11]. Such preformed particle gel is formed by crosslinking polymers under shear flow and expected to control water mobility in order to improve sweep efficiency and reduce water permeability as water production control. They presented several advantages of the microgel, including their quasi-insensitivity to PH, salinity, temperature and shear stress. It was also reported that the microgel had good thermo-stability and good propagation ability in porous media [12].

The number of micrometer-size PPG field applications are less comparing to the number of mm-size PPG applications [10]. Zaitoum et al. reported a field test of micorgel. During the treatment, gel injection volume was cut half of their plan because of an unexpected high injection pressure. However, the water-to-gas ratio of this well was dropped and 25% more gas was produced after treatment. In addition, the sand production of the candidate well was also well controlled after the gel treatment [13].

Submicro- and nanometer-size preformed particle gels. Brightwater[®], a type of submicron sized of gel particles was reported by Nalco Company, ChevronTexaco and BP in 2002[14]. The gel is used to treat matrix problems because it can penetrate into porous media. The particles are initially in the range of microsize when pumping and can expand from 4 to 10 times under reservoir temperature when it is delivered into the in-depth of a reservoir, as shown in like Figure 2.3.

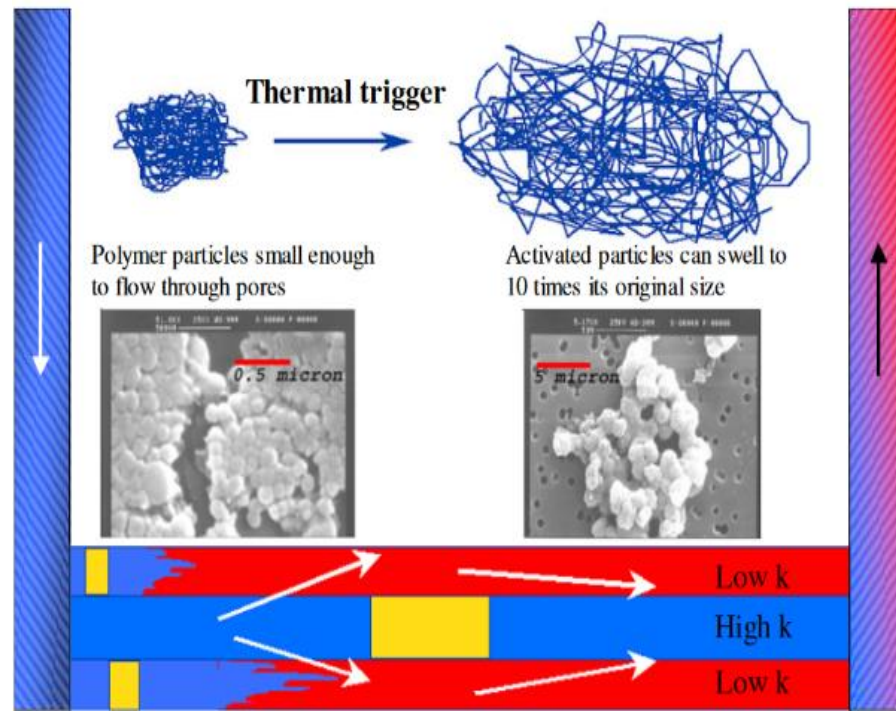


Figure 2.3. Mechanism of conformance control using Brightwater® [14]

Brightwater® has been tested for the first time in Minas field, Indonesia in 2001. Unfortunately, the oil increase is relative low [15]. In another field test in Alaska in 2004, it was predicted that the oil recovery increase from 50,000bbl to 250,000bbl. In fact, over 60,000 bbl oil recovery was increased in the first 4 years after the treatment[16].

2.1.4. Disproportionate Permeability Reduction (DPR) of Gel Treatment.

Disproportionate permeability reduction (DPR) is defined as the treatment process in which the permeability of the medium to one fluid is reduced to a greater extent than the permeability of the medium to another fluid [17]. This is desirable in conformance control since the main goal of gel treatment is to reduce produced water and increase oil

recovery. The ideal DPR technology in conformance control process should not reduce oil permeability at all and not promote oil production reduction in a post-treatment.

However, all known DPR agents reduce oil permeability [18]. Liang et al. list several possible mechanisms for DPR, including: a) the similarity of density between gel and formation brine may result in more particles existing in water phase; b) polymer may lubricate the flow in porous media; c) shrinking and swelling of gel particle would make more pathway for oil; d) residual oil drop would reduce the effective pore size to water flow but not oil flow; e) water and oil may have segregated pathways. As a result, if water-base gel flow through the pathway of water, then the pathway of oil would be remain connected [19].

2.2. PREVIOUS LAB WORKS WITH NANO-SPHERE OR MICRO-SPHERE USING NON-FRACTURE MODEL

To study the transportation and retention of larger particle like millimeter-size particle gel, fractural is a common model in researches. However, to study the properties of nanometer-size and micrometer-size particle gel, homogenous core with a lower permeability is always a good candidate for core flooding experiments.

Resistance factor, residual resistance factor and plugging efficiency are three major terms used to evaluate gel treatment. Resistance factor is defined as the ratio between water mobility and gel mobility. Mathematically, as shown in Equation (1), it could be calculated as the ratio between gel injection pressure and pre-treatment water injection pressure. Residual resistance factor is defined as ratio between water mobility before and after gel treatment. As shown in Equation (2), it could be calculated as the injection pressure ratio between post-treatment water flooding and pre-treatment water

flooding. As shown in Equation (3), plugging efficiency reflect the permeability reduction rate.

$$F_r = \frac{\lambda_{water}}{\lambda_{gel}} = \frac{\frac{k_w}{\mu_w}}{\frac{k_g}{\mu_g}} = \frac{\Delta P_g}{\Delta P_w} \quad (1)$$

$$F_{rr} = \frac{\lambda_{water-before}}{\lambda_{water-after}} = \frac{\Delta P_{after}}{\Delta P_{before}} \quad (2)$$

$$\eta = \frac{K_{before_treatment} - K_{after_treatment}}{K_{before_treatment}} = 1 - \frac{1}{F_{rr}} \quad (3)$$

As shown in the equations above, F_r , F_{rr} and η are residual factor, residual resistance factor and plug efficiency, respectively. λ , k , ΔP and μ means mobility, effective permeability, injection pressure and viscosity, respectively.

Almohsin et al. test the transportation of microgel with diameters ranging from 100 to 285 nanometer in sandstone porous media[20]. Experiments setup is shown in Figure 2.4. When the permeability is 1 Darcy, residual resistance factors are from 2.7 to 4.4 times with different injection velocity. When core samples have lower permeability, residual resistance factors become higher. When rock permeability is 41 mD, residual resistance factors could be hundreds. Based on this study, Goudarzi et al. contained oil phase in core flooding experiments [21]. Oil recovery was improved from 40% to 60% by gel treatment. In the simulation work based on such experiment, on the other hand, oil recovery increase 7%.

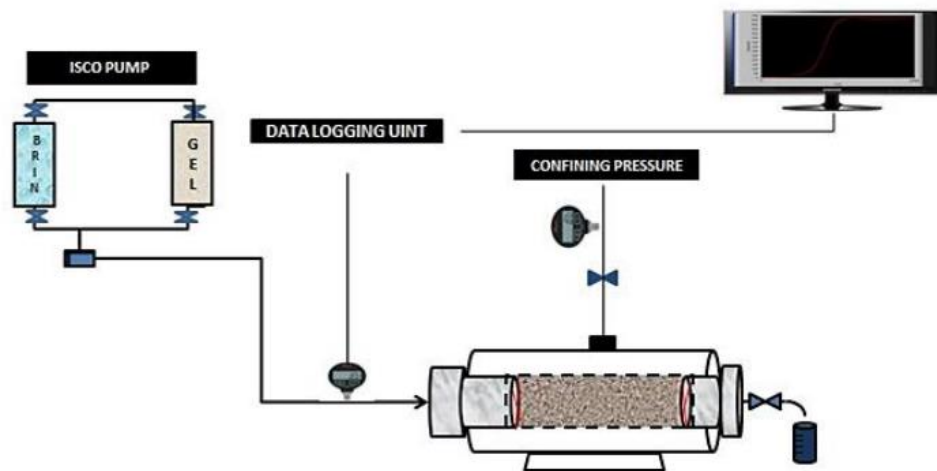


Figure 2.4. Core flooding experiments setup [20]

Dupuis et al. tested SMG (small microgel) in sandstone porous media with residual oil. Meanwhile, there were multiple gel injections with different microgel concentration (lower concentration microgel were injected first). Results showed both resistance factor and residual resistance factor increased with an increase of gel concentration and a decrease of flow rate[22]. In addition, Dupuis et al. studied on SMG's retention in porous media. There were 42 mg gel suspended in porous media after every grams injection[23].

Other than natural cores, there are also some studies using sand packs and artificial cores. Salehi et al. tested Brightwater[®] nanogel using slim tubes with sand inside. Different from Microgel, such nanogel injection pressure was only slightly higher than pre-treatment water flooding pressure because particles will swell with high temperature only. After heating the sand pack model, post-treatment water injection pressure had a significant increase. After breakthrough, injection pressure will drop and

be stable in the end. With a 5,000 ppm nanogel concentration and 5 Darcy permeability, residual resistance factor could be from 2 to 12. With a 10,000 ppm nanogel concentration and lower permeability (from 300 to 1,200mD), residual resistance factor could be from 10 to 50 [24]. Fabbri et al. injected same nanogel into sand pack with a permeability of 7.3 Darcy. After injecting 2.3 pore volumes of 2,300 ppm nanogel, residual resistance factor was just 1.1. Even after heating the sand pack for 41 days with a temperature of 50 Celsius degree, residual resistance factor was still only 1.3 [25].

Lei et al. and Yao et al. studied factors that have influences upon micrometer-size sphere plugging in sand packs. Residual resistance factor would increase with higher sphere concentration and lower injection rate. When the size contrast ratio between sphere and porous media was being increased, residual resistance factor would increase first and then decrease, which approved that there is an appropriate size match between sphere and porous media. Meanwhile, brine component and addition of polymer influence plugging efficiency as well [26, 27]. Song et al. injected nanometer-size sphere into sand packs with different permeability (0.42, 1.7 and 4.8 Darcy). After injecting sphere along with reservoir water, residual resistance factor in lower permeability model was the highest [28].

Zhang injected one pore volume of sphere dispersion (concentration not mentioned) into cemented quartz cores. With the best match between sphere size and permeability, residual resistance factor was 50 [29]. Zhang et al. measured injection pressure in multiple spots of experimental model. Pressure of each spot increase successively, which rule out the possibility of face plugging during gel injection[30].

Lin et al. and Lu et al. studied transportation mechanism of micro-sphere and nano-sphere with filter membrane by recording filtration volume under constant pressure [31, 32].

Vide supra, in most previous work, core models were assumed homogeneous. Imqam et al. studied on non-crossflow heterogeneous model using 75-90 microns sized PPG and sand packs with different permeability [33] Figure 2.5 shows the experiments setup.

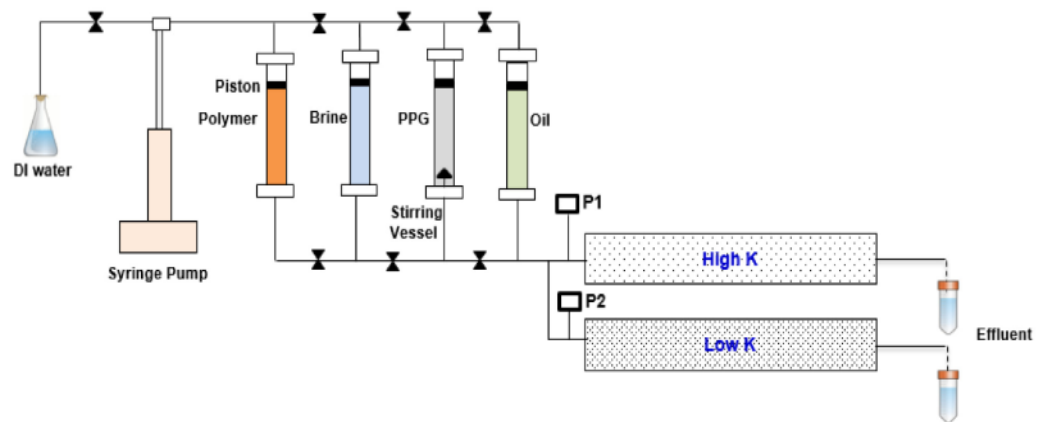


Figure 2.5. Experiments setup of non-crossflow heterogeneous model [33]

When the permeability contrast ratio is 4 (21.7 Darcy and 6.2 Darcy), oil recovery factors in lower permeability sand pack and higher permeability sand pack were 20% and 80% respectively during pre-treatment water flooding. After PPG treatment, the recovery factor in lower permeability sand pack increased approximately 70% total. The oil recovery improvements with different models are shown in Table 2.1. What is more,

before PPG treatment, there were less than 5% of total injection water going through the lower permeability sand pack. After PPG treatment, 63% of total injection water went through the lower permeability sand pack. When permeability contrast ratio is higher (20 and 44), profile improvements were even better, as shown in Table 2.2.

Table 2.1. Oil recovery improvement using different models [33]

Permeability Contrast Ratio	Permeability, Darcy	Incremental Oil Recovery Ratio		
		Before PPG	During PPG	After PPG
4	High 21.7	80	80.1	80.2
	Low 6.2	20	31.7	92.1
20	High 22.4	74	74	74
	Low 1.1	1.9	1.9	60
44	High 22.1	52.2	52.2	53
	Low 0.5	0.9	0.9	36

Table 2.2. Injection profile improvement using different models[33]

Permeability Contrast Ratio	Permeability, Darcy	Injection Profile,%		Incremental Injection Profile in Low Permeability Cores
		Before PPG Injection	After PPG Injection	
4	High, 21.7	90	34	
	Low, 6.2	5	63	12
20	High, 22.4	83	80	
	Low, 1.1	0.5	15	30
44	High, 22.1	88	55	
	Low, 0.5	0.1	33.6	336

3. PAM MICROGEL

3.1. EXPERIMENTAL DESCRIPTION

The following sections introduced the materials, setup, workflow and the plan of this work.

3.1.1. Materials. Major materials used in this study are listed below.

Microgel: Polyacrylamide (PAM) gel is an acrylamide based, crosslinked hydrogel. As shown in Table 3.1, there are four types of PAM microgel samples. They are synthesized by water, AM (Acrylamide), AA (acrylic acid) and MBAA (N, N'-Methylenebisacrylamide). MBAA is served as crosslinker and is the major different among all types of PAM microgels.

Table 3.1. Component and proportion of each PAM samples

PAM#	Water/g	AM/g	AA/g	MBAA/g
A	15	10	5	0.0375
B	15	10	5	0.0075
C	15	10	5	0.00075
D	15	10	5	0.00025

Core samples: core samples being used in all experiments are Berea sandstone™ which have been believed to be the best sandstone for core flooding experiments. Berea Sandstone™ is a kind of sedimentary rock, which grains are predominantly sand-size. Such rocks are composed of quartz held by silica. Berea Sandstone™ also have relatively

high porosity and permeability, which make it a good reservoir rock[34]. Each core sample has a diameter of 2 inches and a length around 5 inches.

Brine: 1 weight percent NaCl solution.

Oil: light mineral oil from Fisher Scientific. Such oil has a viscosity of 33.5 cSt (33.5cP).

3.1.2. Experimental Setup. The experimental setup in this study is depicted in Figure 3.1.

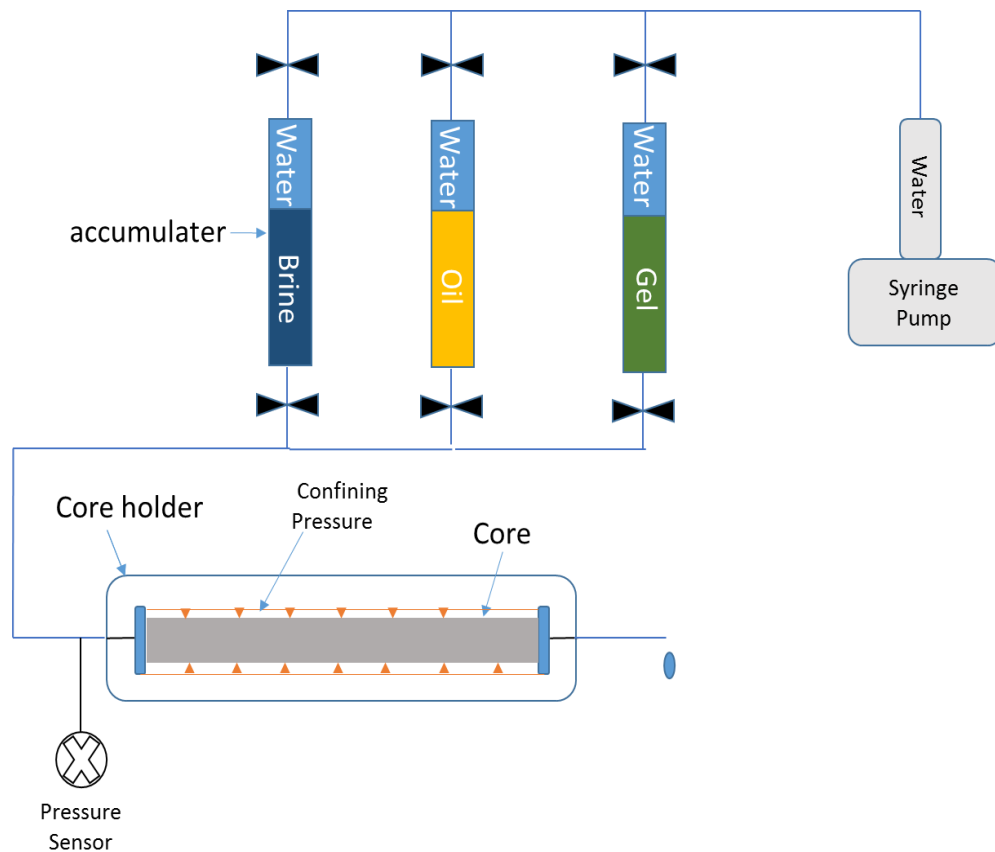


Figure 3.1. Schematic diagram of setup of experiments

As shown in the figure, the core holder could hold a core with a diameter of 2 inches and a length between 4 and 5 inches. A syringe pump is used to inject brine, oil and micro gel from accumulators into core samples. There is a piston inside accumulators. When water is being injected, it will push the piston in order to inject oil, brine or gel into the core samples. The confining pressure system is used to insure injection fluid go through core samples. The confining pressure is normally set at 400 psi above injection pressure. There is a pressure sensor connected in front of core holder, which could be used to collect the injection pressure data. There are also test tubes being kept at the outlets of core holder to collect effluents.

3.1.3. Experimental Procedures. The flowing subsections are the briefly explanation of procedures used to perform experiments. Figure 3.2 shows the flow chart of experiments.

Preparation and saturation of core sample. After drilling and incision core samples from blocks of rock, core sample were put into oven with 65°C for enough time until there is no water inside porous media. Then the sample was vacuumed for at least 6 hours and saturated with brine. The weight difference between dry sample and saturated sample is the weight of brine inside the sample. Pore volumes could be calculated with brine density.

Permeability measurement. 1wt% NaCl brine was injected into core samples at 5 different injection velocities. Pump flow rates and corresponding velocities being used in experiments are shown in Table 3.2. According to Darcy's law, permeability can be calculated with injection pressure data.

The effluents from next four step were collected to determine the initial oil saturation and oil recovery factors. In two oil injections and brine injections, five flow rates were used as shown in Table 3.2. All injections were stopped only with a stable injection pressure and negligible water cut (for oil saturation step) or oil cut.

Table 3.2. Injection velocity and their corresponding pump rates conversion table

Pump flow rate, ml/min	Injection velocity, ft/day
1	2.4
1.25	3.0
1.5	3.6
1.75	4.2
2	4.8

Oil saturation. Mineral light oil was injected into core samples at a flow rate of 1ml/min until injection pressure reach stability. Then use four more flow rates to get the injection pressure with different flow rates. Effluents were collected to calculate initial oil saturation.

First water flooding. Brine would be injected following oil saturation at five flow rates.

Microgel treatment. Microgel particle dispersion was injected into samples at 1 ml/min after fully dispersed in 1wt% NaCl brine with a concentration of 2,000 ppm.

Second water flooding. After microgel injection, another water flooding will be performed to get the residual resistance factor to water.

Second oil injection. Light oil would be injected again to determine the residual resistance factor to oil.

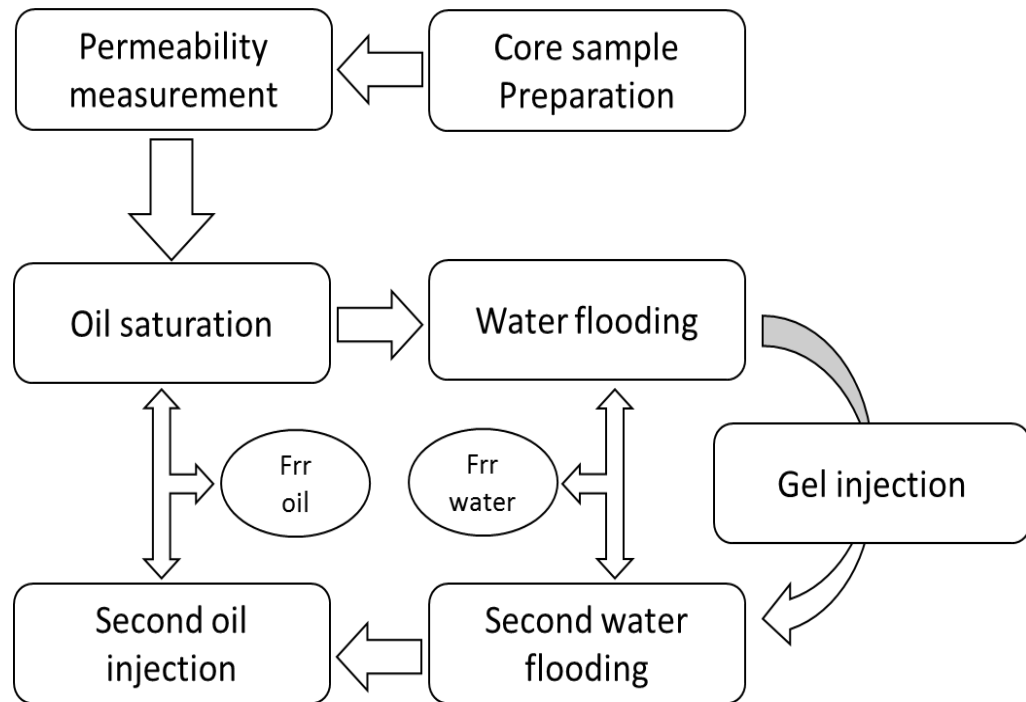


Figure 3.2. Experimental flow chart

Practically, microgel particle will increase oil recovery by conformance control in heterogeneous reservoir. However, all core samples used in this study are homogenous. As a result, such treatment will not increase oil recovery but only decrease

permeability of samples ideally. In fact, there was still oil recovery increment in each experiments, oil recovery data were all recorded and analyzed.

3.1.4. Experimental Plans. Permeability and crosslinker concentration are The variables in this study. To study permeability's impact on microgel treatment, particle #A was used in all three experiments with different permeability core samples. To study the effect of crosslinker concentration, permeabilities of core samples were all in a range of 200mD while different PAM microgels were tested in each experiment. Crosslinker concentrations and particle sizes after swelling in 1wt% NaCl brine are shown in Table 3.3. Particle size data are measured by Dynamic light scattering (DLS), which is a technique in physics that can be used to determine the size distribution profile of small particles in suspension or polymers in solution. However, since the sizes measured by DLS are the hydrolysis dynamic radius, the number will be larger than the true sizes of microgel particles. Before swelling, all particles have a similar diameter around 50 nanometers, which are all measured by scanning electron microscope (SEM).

All experiments performed are listed in Table 3.4. Experiments #1, #2, #4 and #5 were performed with different crosslinker concentration are. In experiments #1, #3 and #6, core samples had different permeabilities.

3.2. RESULTS FROM EXPERIMENTS

3.2.1. The Effect of Permeability on Microgel Treatment. To study the effect of permeability, Gel #A, which has a diameter of 354.2 nanometer after swelling, and three core samples with permeabilities of 262.1mD, 56.8mD and 23.4mD were used in this work. The pore size and particle/porous media size contrast ratio are shown in Table

3.5. With different permeabilities, the estimated diameters of porous media are about 18, 9 and 6 times to particle sizes. The porous media sizes data were calculated with permeability and porosity data by an empirical equation shown in equation (4).

$$k = 20 \times 10^6 d^2 \phi \quad (6)$$

Table 3.3. Crosslinker concentration and particle sizes after swelling

Particle	Crosslinker concentration, ppm	Particle size after swelling, nm(diameter)
A	1248	354.2
B	250	538.6
C	25	615.1
D	8	955.4

Table 3.4. Experiments performed in this study

Experiments	Gel selection	Permeability, mD
1	A	262.1
2	B	264.3
3	A	56.8
4	C	211.0
5	D	191.4
6	A	23.4

Figure 3.3 and 3.4 show gel treatment and second water flooding injection pressure plots with a flow rate of 1ml/min, respective. Y axis in these plots are resistance factor, which is the injection pressures ratio between gel treatment and first water flooding at 1ml/min, and residual resistance factor, which is the injection pressures ratio between second water flooding and first water flooding at 1ml/min(the calculation is based on equation 1 and 2).

Table 3.5. Pore sizes and particle size contrast
(Size contrast ratio: particle diameter/porous media diameter)

Experiment	Permeability, mD	Porosity, %	Porous media diameter , μm	Size contrast ratio
1	262.1	21.08	6.33	1: 17.9
3	56.8	18.32	3.16	1: 8.9
6	23.4	18.13	2.04	1: 5.8

As shown in Figure 3.3 and 3.4, during gel treatment, injection pressure increase slowly at first. After reach a maximum value, pressure would drop and reach a stable value in the end.

Similar trend happened during second water flooding as well. The injection pressure goes to the peak immediately following the start of injection and then drop to a stable value.

The pressure drop is a result of gel strength under high injection pressure. When injection pressure reaches a higher range, microgel particle is no longer strong enough anymore. Therefore, injection flow would partially break through and result in a pressure drop.

Figure 3.5 shows the peaks of resistance factors during gel treatment and the resistance factors after a stable injection pressure. Figure 3.6 shows the peaks of residual resistance factors during second water flooding and the residual resistance factor after a stable injection pressure. The flow rate was all 1ml/min. As shown in the figures, when permeabilities were lower, both peaks and stabilized value of RF and RRF are lower.

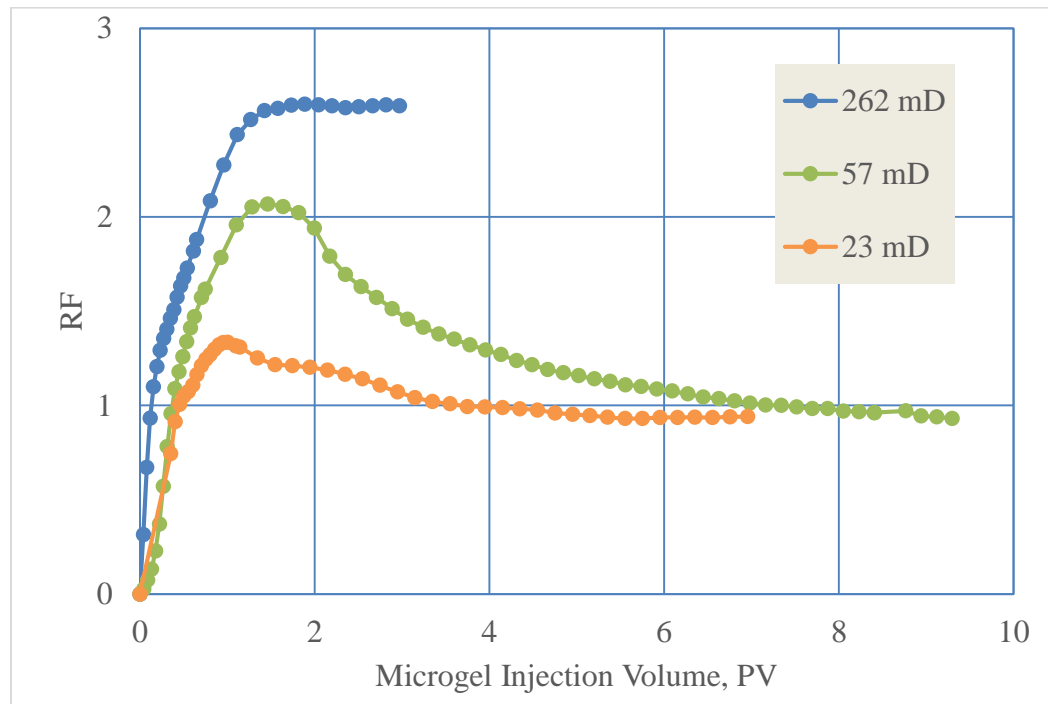


Figure 3.3. Gel injection pressure plots with different permeability

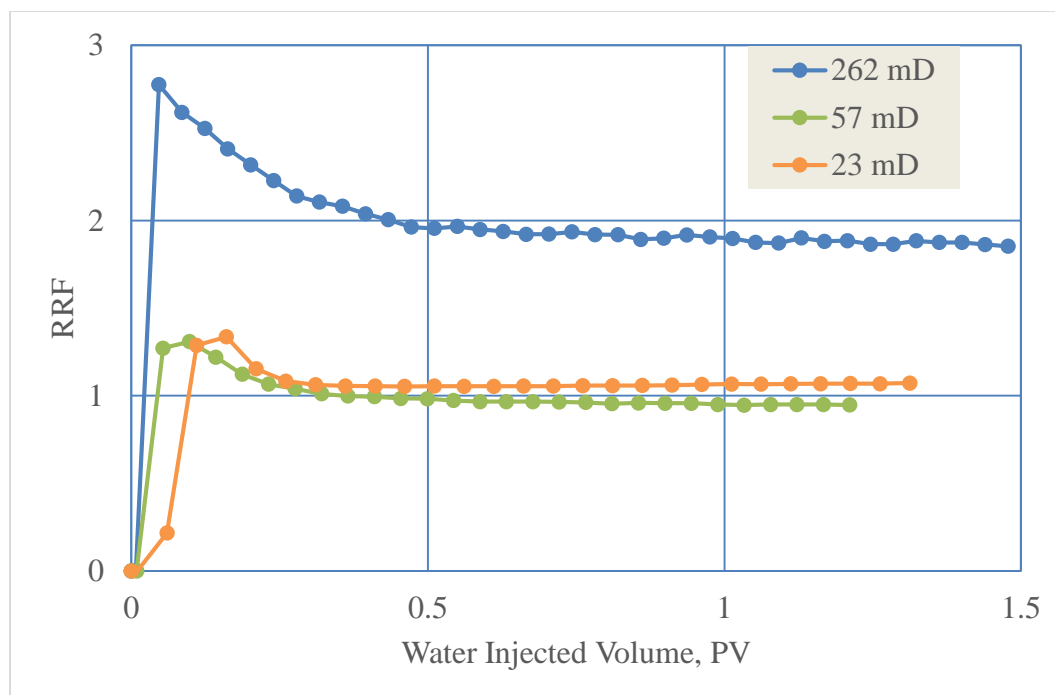


Figure 3.4. Second water flooding pressure plots with different permeability

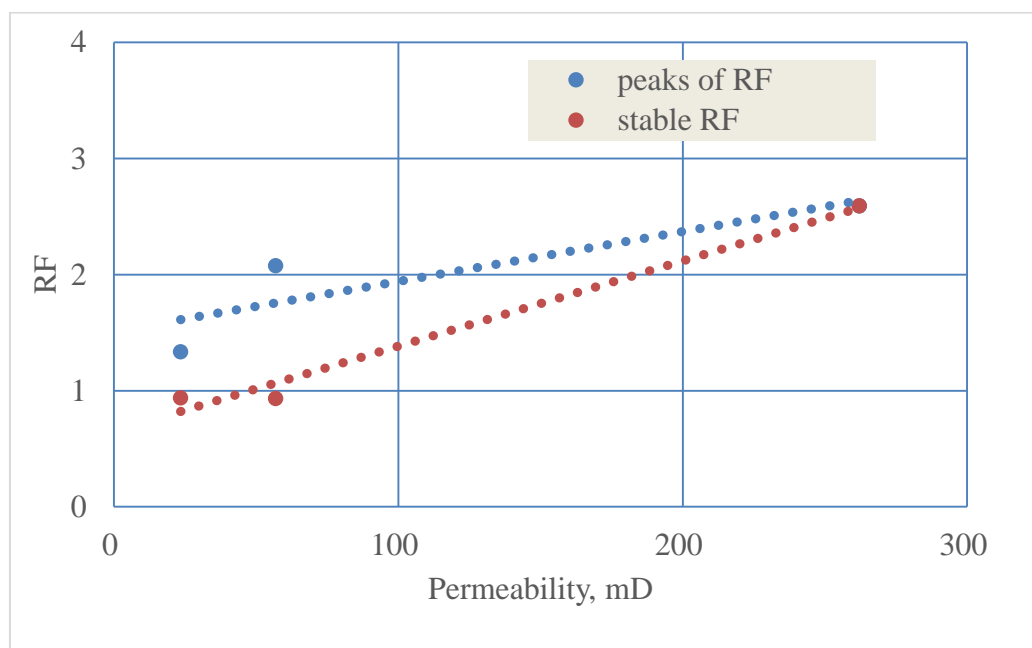


Figure 3.5. Resistance factor and the peaks of gel injection and second water flooding

Figure 3.7 and Figure 3.8 show the residual resistance factors to water and oil at different flow rates. When performing experiments using the core sample with the highest permeability, RRF to water is the highest. Meanwhile, with lower permeability, RRF to oil is higher than RRF to water. Only exception is the experiment using highest permeability core sample, where RRF to oil is lower than RRF to water.

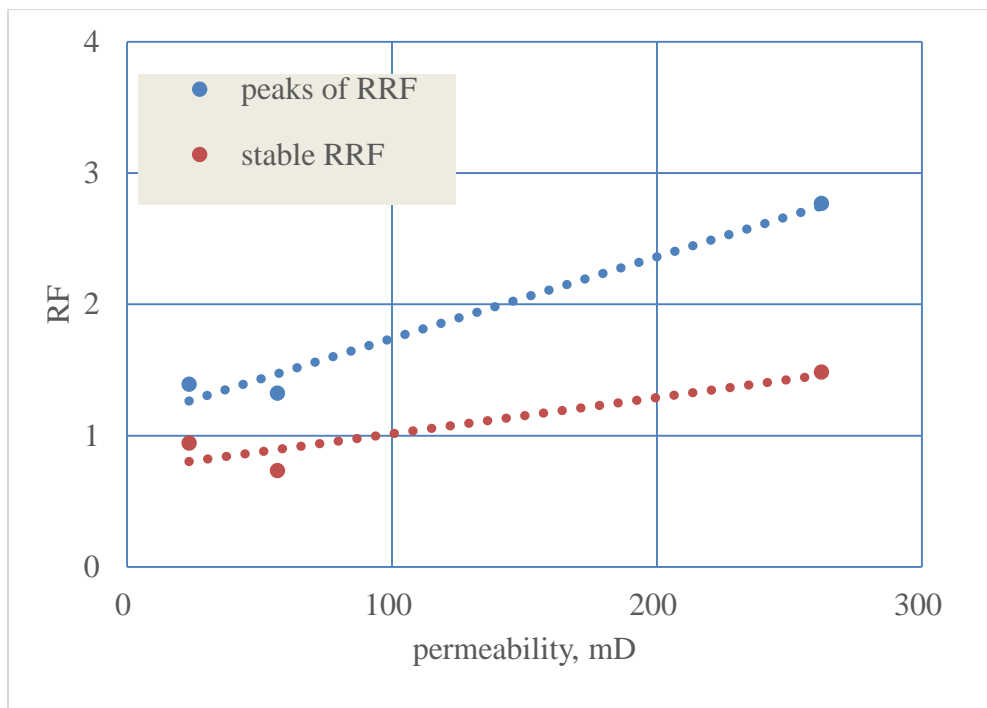


Figure 3.6. Residual resistance factor and the peaks of second water flooding

Figure 3.9, 3.10 and 3.11 show the oil recovery factor plots at different stages of each experiment. Y-axis of each plots are scaled up for emphasizing oil recovery improvement during gel treatment and second water flooding. As shown in the plots,

during gel treatment, oil recovery increased after a certain amount of injection. Compare to oil recovery increment during gel treatment, there were less oil recovery increment during second water flooding.

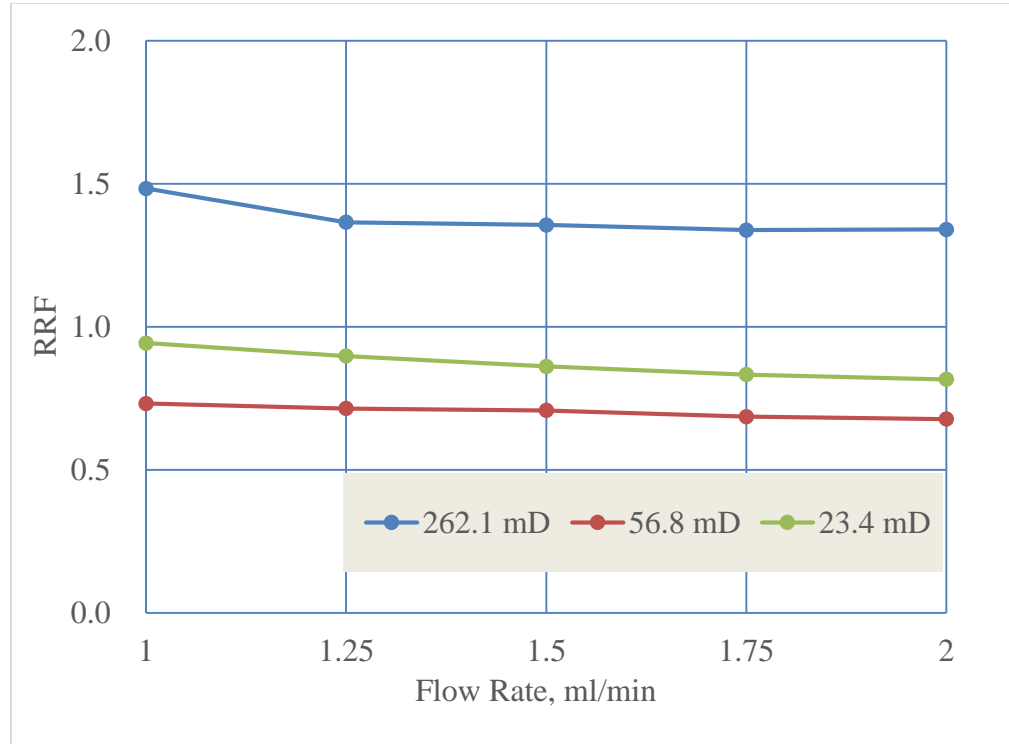


Figure 3.7. Residual resistance factor to water

Table 3.6 summarize the oil recovery factor data in all three experiments. After first water flooding, oil recoveries were all around 45%. When high permeability core sample being used, oil recovery improved by gel treatment is the lowest. When permeability is lower, oil recovery improvement could be higher than 5%. It shows that lower permeability is favorable for improving oil recovery.

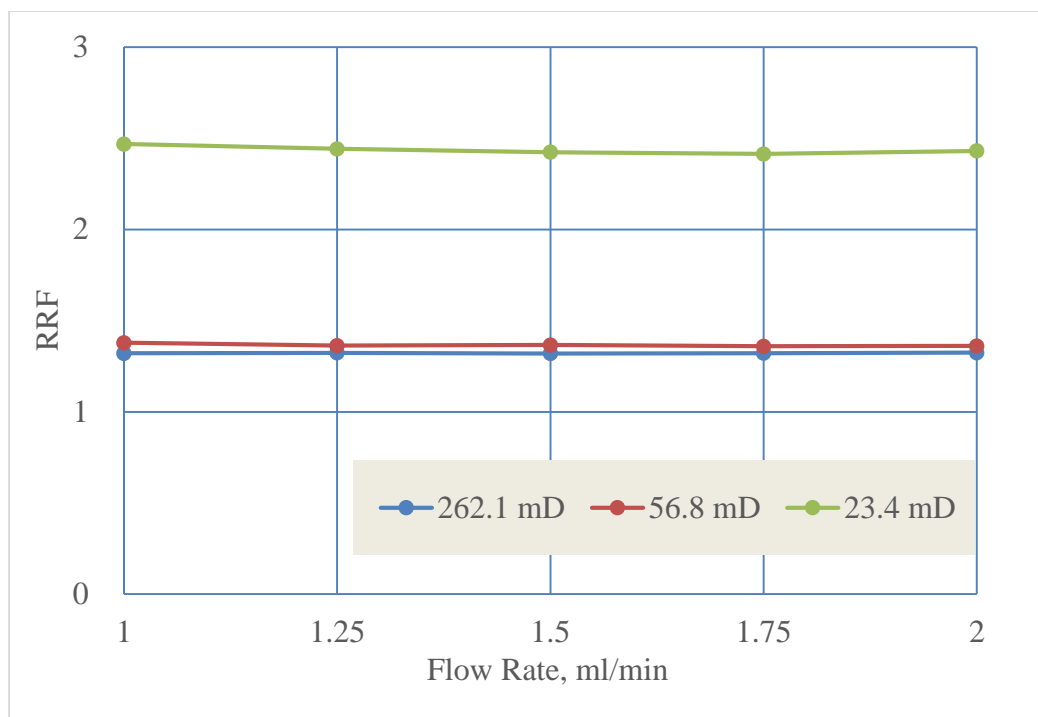


Figure 3.8. Residual resistance factor to oil

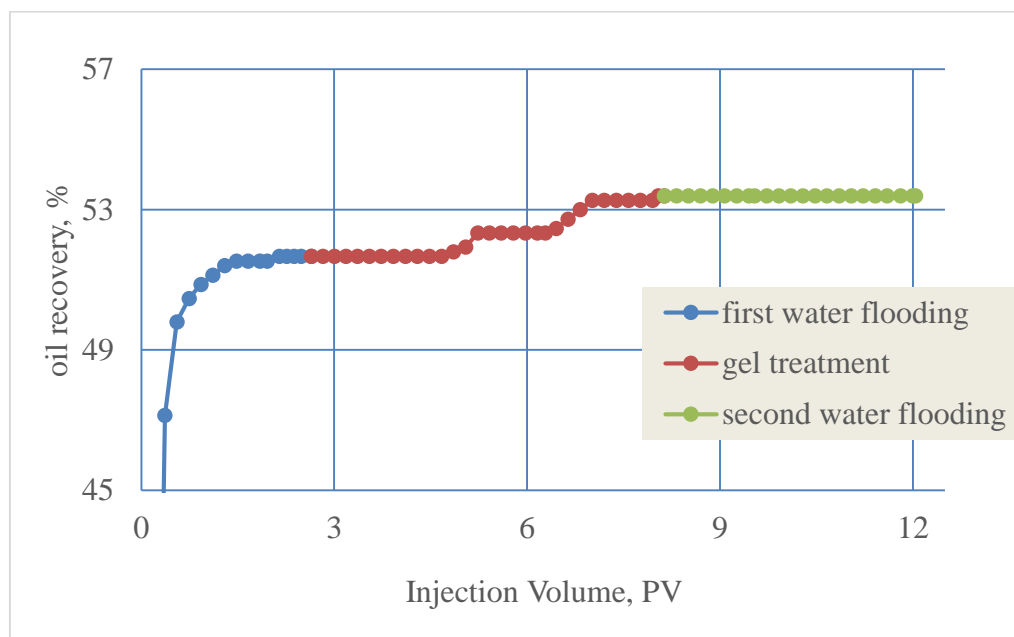


Figure 3.9. Oil recovery plot of experiment #1

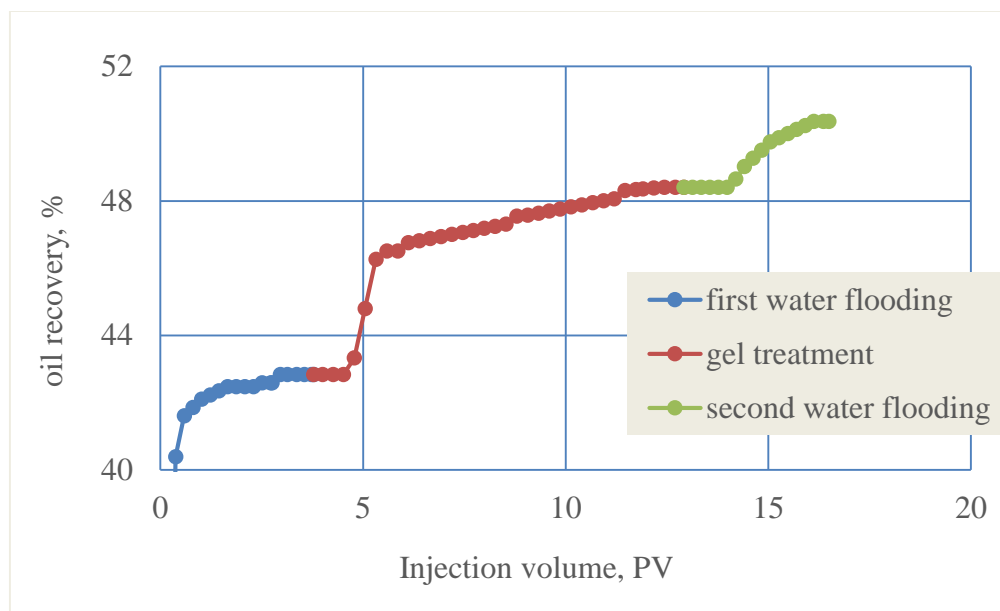


Figure 3.10. Oil recovery plot of experiment #3

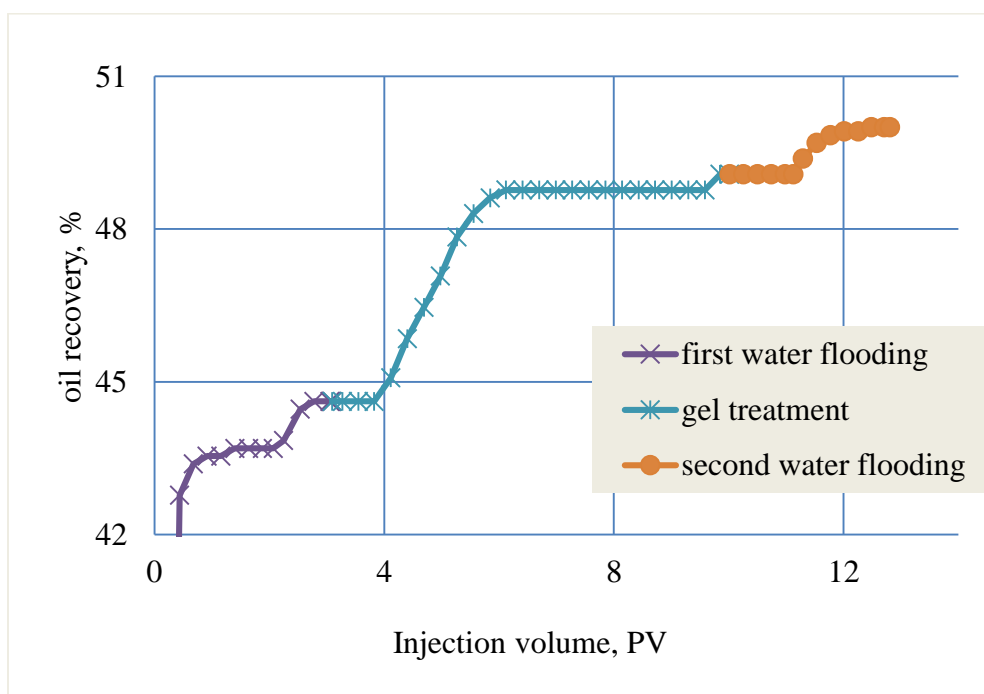


Figure 3.11. Oil recovery plot of experiment #6

Table 3.6. Oil recovery factors in different stages
(W.F.: water flooding; Gel: gel treatment; 2nd W.F.: second water flooding)

Experiment	Permeability, mD	Oil recover factors, %			Oil recovery increment, %
		W.F.	Gel	2nd W.F.	
1	262.1	45.27	1.73	0.00	1.73
3	56.8	47.17	6.13	2.16	8.29
6	23.4	44.62	4.46	0.92	5.38

3.2.2. The Effect of Crosslinker Concentration on Microgel Treatment. As mentioned earlier in Section 3.1.4, four different crosslinker concentrations were used during microgel synthesis. Core samples' permeabilities, porous media sizes and the contrast ratios to particle sizes are shown in Table 3.7. Pore sizes are roughly 18, 12, 9 and 6 times to particle sizes in experiments #1, 2, 4 and 5, respectively.

Figure 3.12 and 3.13 shows gel treatment and second water flooding injection pressure plots at 1ml/min. Figure 3.14 shows the peaks of resistance factors during gel treatment and the resistance factors after a stable gel injection pressure. Figure 3.15 shows the peaks of residual resistance factors and the stabilized residual resistance factor during second water flooding. The flow rate was also 1ml/min. There is a clear trend that with less crosslinker concentration, which could result in larger particle size, both resistance factors and residual resistance factors are higher. Meanwhile, maximum injection pressures during both gel treatment and second water flooding are also higher with a decrease in crosslinker concentration.

Figure 3.16 and 3.17 show the residual resistance factors to oil and residual resistance factors to water at all five flow rates. With less crosslinker concentration, micro particle gel has better plug efficiency. All particle but #B have a higher RRF to water compare to the RRF to oil, which shows a good disproportionate permeability reduction(DPR).

Table 3.7. Particle/pore size contrast ratio
(Size contrast ratio: particle diameter/porous media diameter)

Experiment	Microgel	Crosslinker concentration, ppm	Particle diameter, nm	Size contrast ratio
1	A	1248	354.2	1:17.9
2	B	250	538.6	1:11.7
4	C	25	615.1	1:9.1
5	D	8	955.4	1:5.7

Figure 3.18, 3.19 and 3.20 are the oil recovery plot of experiment #2, #3 and #5. The plot of experiment #1 shows in Figure 3.9. Plots are also scaled up for emphasizing oil recovery increase.

Oil recovery factors data are summarized in Table 3.8. As shown in the table and plots, when the crosslinker concentration is lower, which means particles sizes are bigger, there would be a higher oil recovery increment.

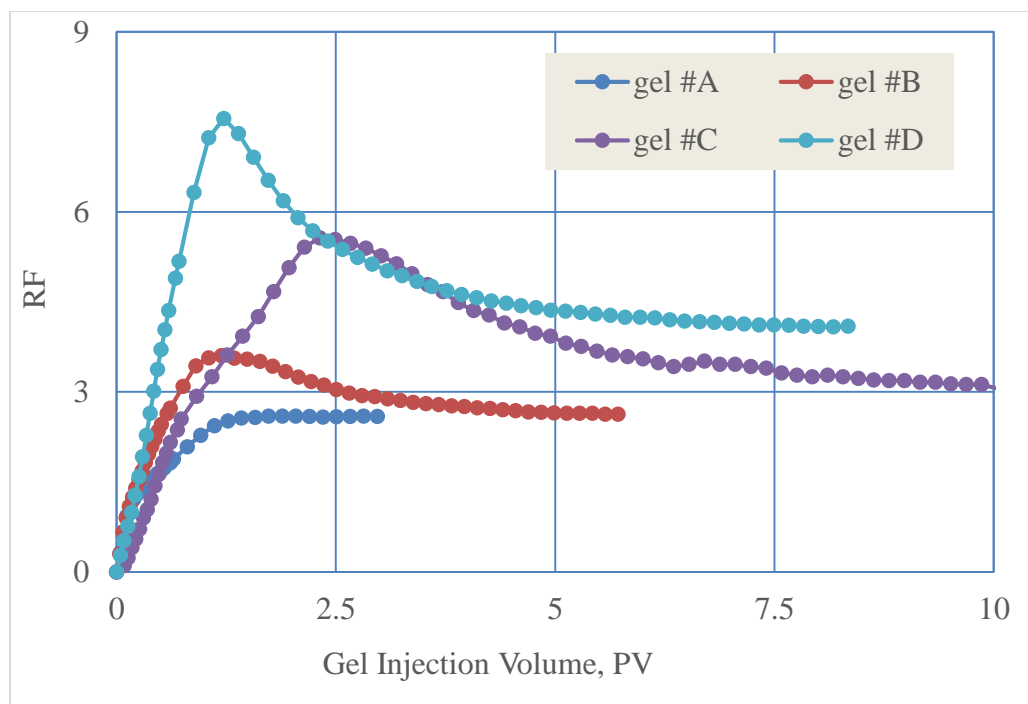


Figure 3.12. Gel injection pressure plots with different crosslinker concentration

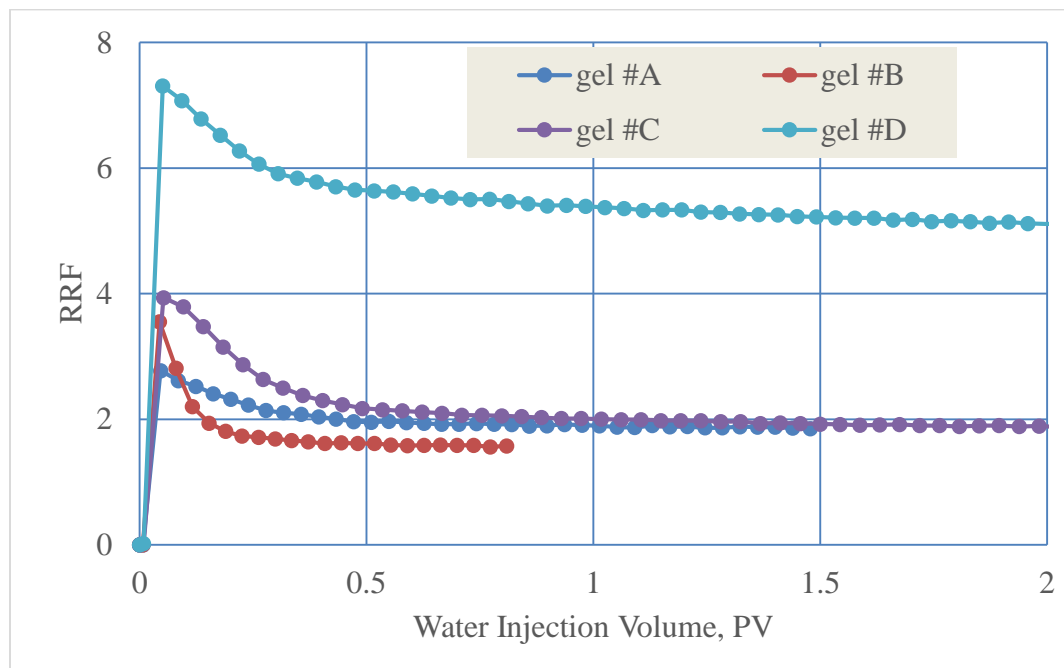


Figure 3.13. Second water flooding pressure plots with different crosslinker concentration

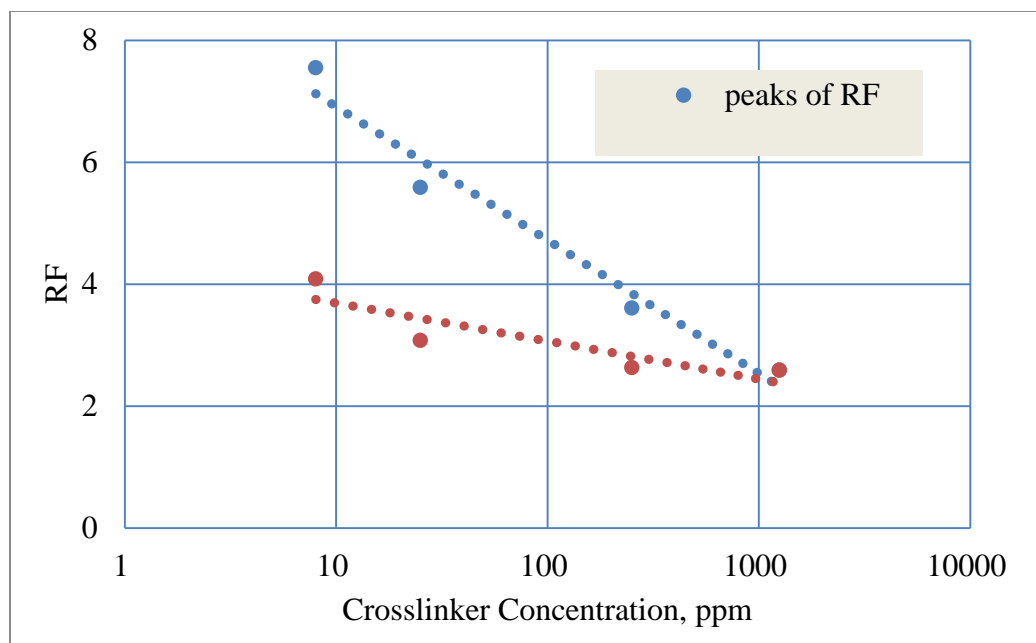


Figure 3.14. Resistance factor and the peaks of gel injection pressure

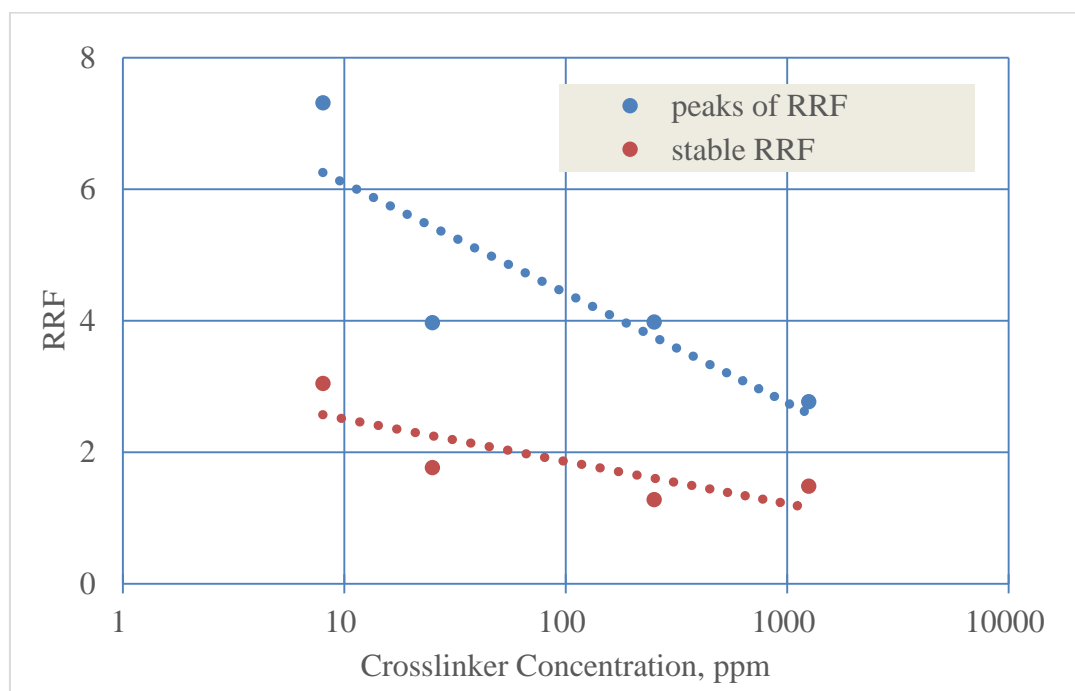


Figure 3.15. Residual resistance factor and the peaks of gel injection pressure

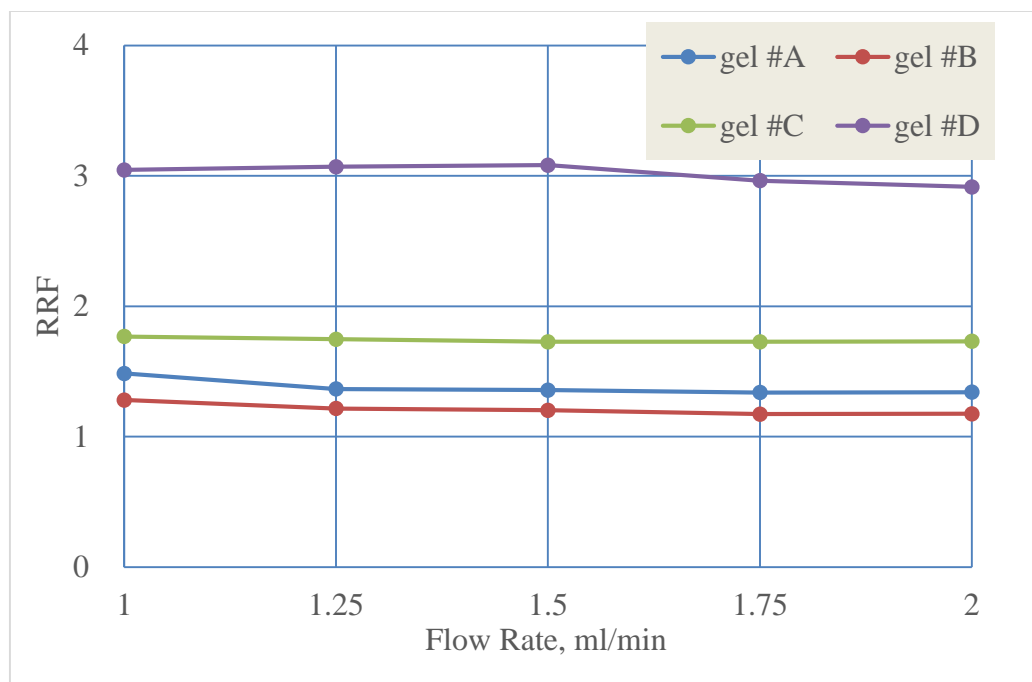


Figure 3.16. Residual resistance factor for water

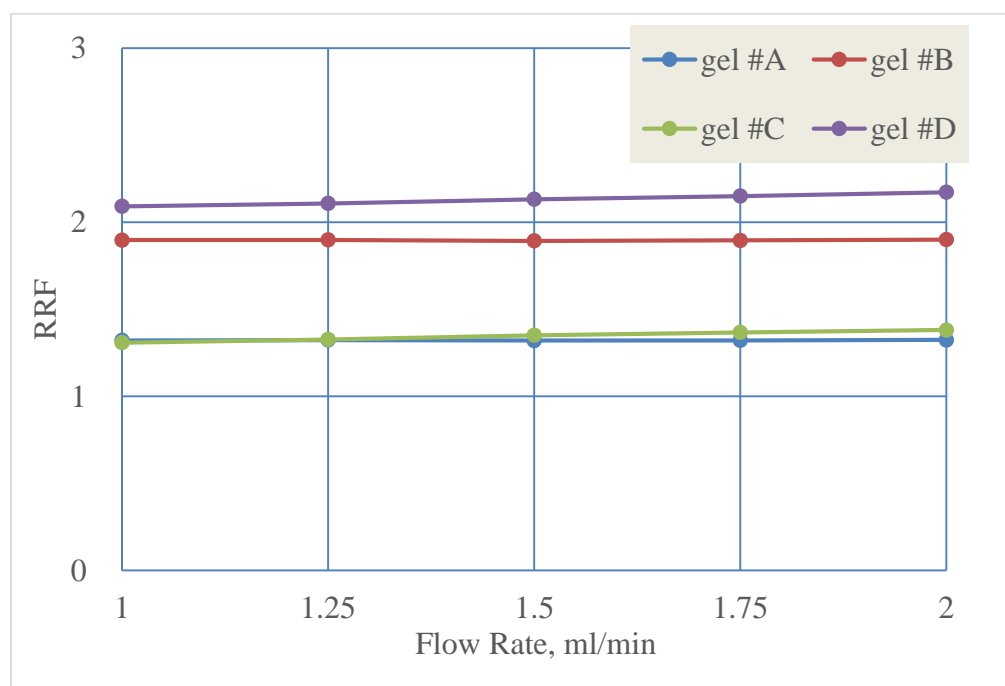


Figure 3.17. Residual resistance factor for oil

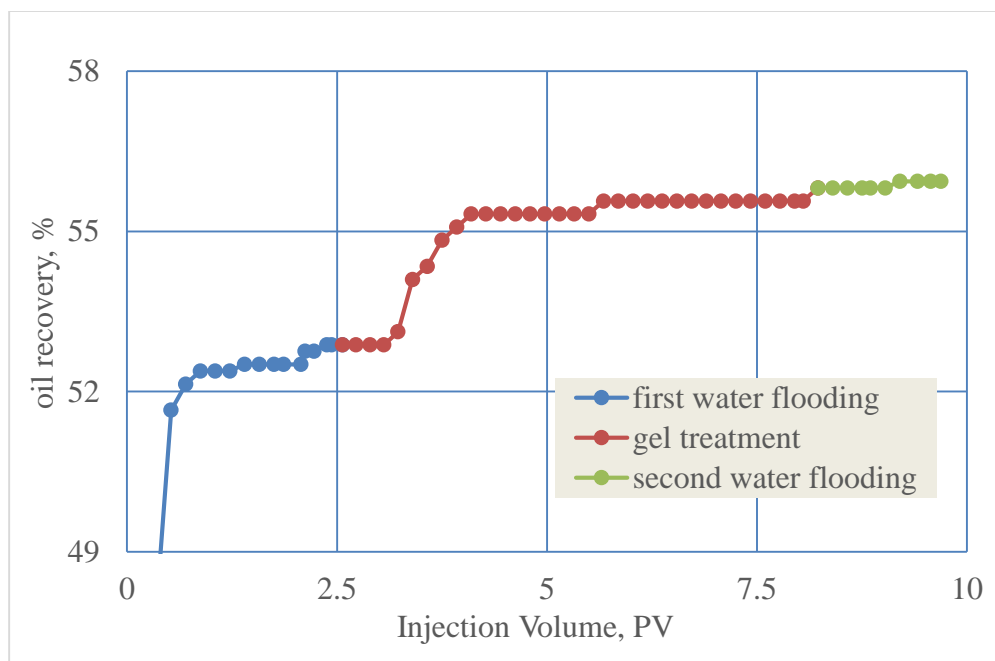


Figure 3.18. Oil recovery plot of experiment #2

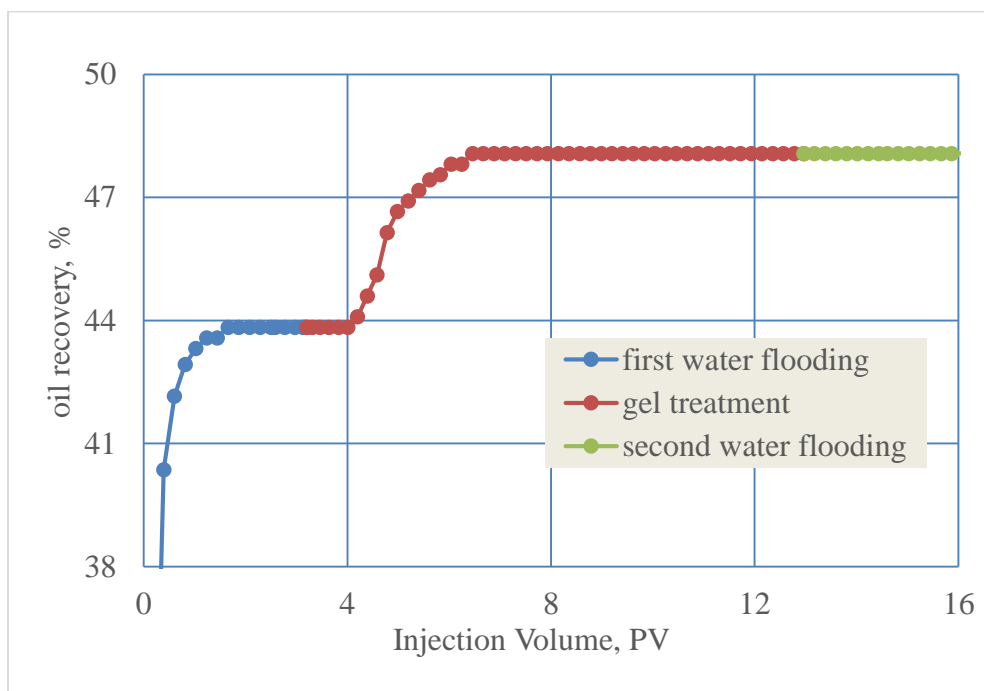


Figure 3.19. Oil recovery plot of experiment #4

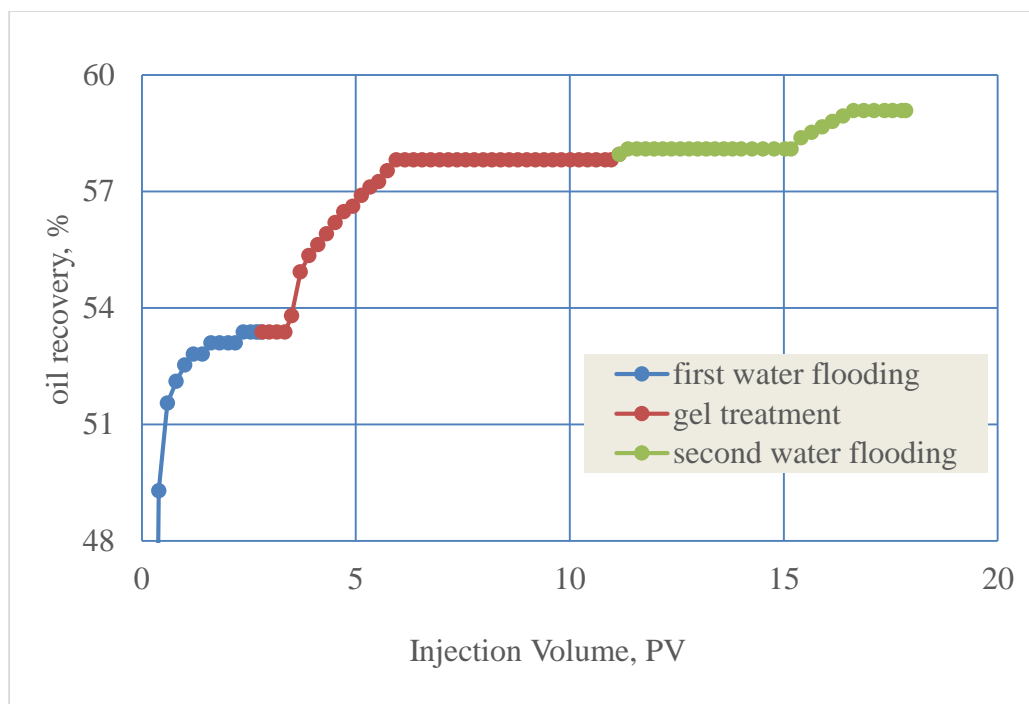


Figure 3.20. Oil recovery plot of experiment #5

Table 3.8. Oil recovery factors in different stages
(W.F.: water flooding; Gel: gel treatment; 2nd W.F.: second water flooding)

Experiment	Particle size, nm	Oil recovery factors, %			Oil recovery increased, %
		W.F.	Gel	2nd W.F.	
1	354.2	45.27	1.73	0.00	1.73
2	538.6	52.88	2.69	0.37	3.06
4	615.1	43.83	4.24	0.00	4.24
5	955.4	53.38	4.44	1.27	5.70

3.3. DATA ANALYSIS AND DISCUSSION

The results indicated that within a same permeability range, microgels with larger swollen ratio have better performance as both oil-displacing agent and plugging agent. From other studies like Almohsin et al. (2014), which is mentioned earlier in Section 2.2, the results showed with higher swollen ratio, microgel particles were weaker since both resistance factor and residual resistance factor became lower. However, in Almohsin et al.'s work, swollen particle sizes were controlled by brine concentration. With lower brine concentration, particle sizes after swelling would be higher. In this study, on the other hand, swollen ratio was controlled by crosslinker concentration and all experiments were done with the very same brine. Moreover, microgel in both studies were different.

When studying the effect of crosslinker concentration, microgel had better plugging efficiency in high permeability core sample, but improved oil recovery better in lower permeability core samples.

The high injection pressure is the reason caused the poor plug efficiency in low permeability core samples. Table 3.9 shows the injection pressures of each experiment in the part of study. As shown in the table, compare to experiment #1, when using lower permeability core samples, injection pressures were much higher. Hence, microgel particles are no longer strong enough under such high injection pressure anymore.

Moreover, comparing experiments #5 and #6, where the contrast ratios are close (5.7 and 5.8), the first water flooding pressure at 1 ml/min are 8.7 psi and 232 psi, respectively. Therefore, RF and RRF in experiment #5 are 4 and 3, while RF and RRF in experiment #6 were both only 0.94.

Table 3.9. Injection pressure at 1ml/min in experiment #1, 3 and 6

Experiment	First water flooding stable pressure, psi	Gel injection break through pressure, psi	Stable gel injection pressure, psi	Second water flooding break through pressure, psi
1	8.12	21.05	21.05	22.47
3	70.30	145.90	65.50	93.00
6	232.00	309.80	217.90	322.90

Meanwhile, gel treatment improved oil recovery better with a lower crosslinker concentration or in lower permeability porous media, which indicate that a higher particle/pore size contrast ratio (larger particle or smaller porous media) is favorable for gel treatment to improve oil recovery.

Figure 3.21 shows the residual resistance factors to both fluids in each experiment. As shown in the figure, residual resistance factors to water is higher than the residual resistance factors to oil in experiment #1, #4 and #5. Such result shows a favorable disproportionate permeability reduction for improving oil recovery. However, in experiment #2, #3 and #6, the disproportionate permeability reduction is not favorable since residual resistance factors to oil are higher, which indicate that PAM microgel treatment cannot always result in a favorable DPR for improving oil recovery.

Meanwhile, in experiment #3 and #6, residual resistance factors to water were even lower than 1 after microgel treatment. Such phenomenon could be explained with relative permeability theory. As mentioned earlier, there are 8.29% and 5.38% oil increment in the experiments. As a result, with higher water saturation, the relative

permeability to water become higher and relative permeability to oil become lower. As the definition of relative permeability (Equation 5), if absolute permeability remain same, effective permeability to water would be higher, which cause lower injection pressure. In fact, the absolute permeability would not remain same after gel treatment since it is the goal of microgel treatment to decrease it. But it will explain the low residual resistance factors to water in certain experiments. Meanwhile, microgel with better strength and DPR would be desirable for better microgel treatment.

$$K_{relative} = \frac{K_{effective}}{K_{absolute}} \quad (5)$$

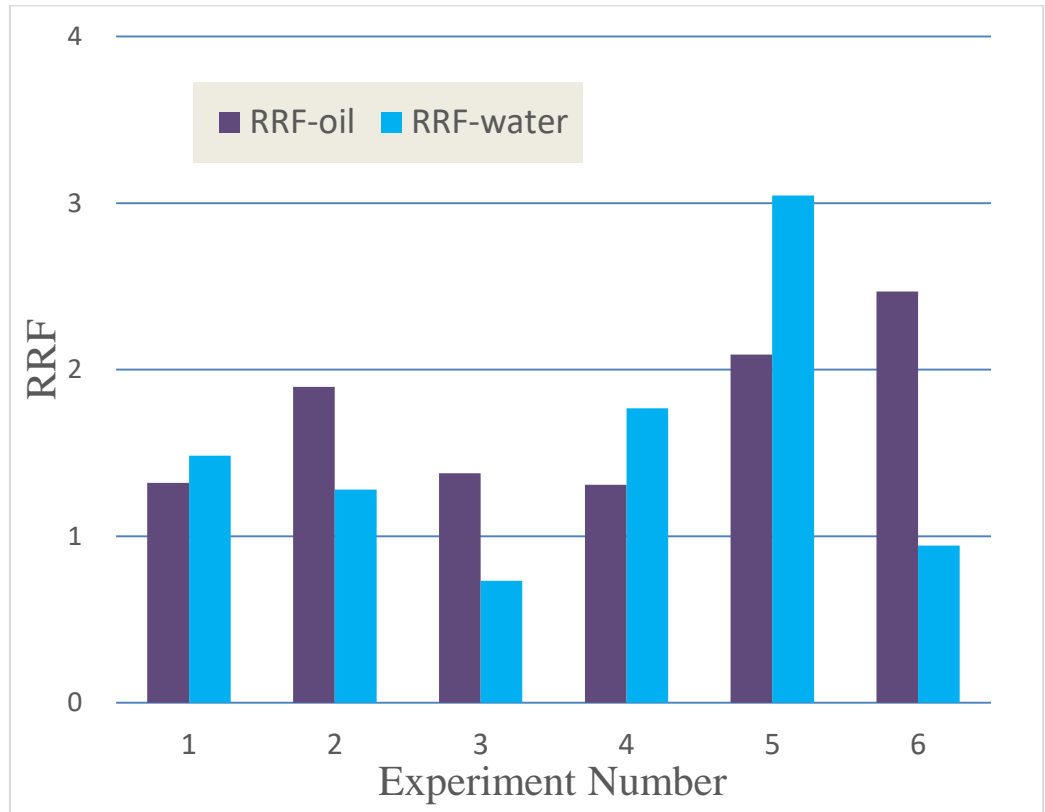


Figure 3.21. RRF at 1 ml/min for oil and water

3.4. SUMMARY

According to the results from experiments that had been done in this section, the following summaries on PAM microgel can be drawn:

1. Within a same permeability range, both resistance factors and residual resistance factors increased when crosslinker concentration decreased.
2. For the microgel synthesized by the same concentration of crosslinker, plugging efficiency become less when permeability was reduced because low permeability rocks required higher injection pressure gradient, which might cause the microgel particles move out of rocks.
3. Even though all the core samples used in the experiments are homogenous, there are still oil recovery increment during and after gel injection. This indicate that other than conformance control, microgel could also increase oil recovery by other mechanisms.
4. Oil recovery increment would be higher in lower permeability porous media or using larger microgel particles. This result indicates that a higher particle/pore size contrast ratio is favorable for improving oil recovery.

4. RE-CROSSLINKED MICROGEL

4.1. EXPERIMENTAL DESCRIPTION

4.1.1. Materials. Oil, brine and core samples used in this work were same as the material used in previous work.

Microgel: Re-crosslinked micro gel particle is a novel type or microgel which could become bigger particle by re-crosslinking under high temperature inside reservoir. Figure 4.1 shows the results of DLS tests on re-crosslinked microgel particles. Y-axis represent distribution while x-axis represent diameters of particles. As shown in figure a, b and c, after being dispersed in 1 wt% NaCl solution, the peak of distribution plot was at 196.6 nanometer. Then, the dispersion was put inside an oven with 65 °C (147°F). After one day, there were two peaks in the distribution plot. The higher peak was at 268.9nm and the lower peak was at 1000nm. After three days, there was only one peak which is at 1041nm. In general, the diameter of most particle changed from 196.6 nm to 1041 nm. Meanwhile, the test was performed twice, as shown in Figure 4.1, the red and green curves means the DLS results of each measurement. In all experiments, Re-crosslinked microgel had a concentration of 3,000 ppm.

4.1.2. Experimental Plans. First, core flooding experiments were performed without oil phase. Then, oil phase was contained in experiments. Because of the different result from two groups of experiments, two more experiments were performed to study the impact of oil phase and oil saturation on core flooding experiments.

4.1.2.1 The plugging ability of Re-crosslinked microgel in single phase condition. In this part, two experiments were performed with water (1 wt% NaCl solution) as the only phase evolved in experiments. The setup of experiments is similar to

the one mentioned in Section 3.1.2 and Figure 3.1, where the only difference is the size of core samples. The diameter of core samples was 1 inch and the length was 2.5 inches.

Table 4.1 shows the permeabilities, estimated diameters of porous media and its contrast ratios with particle size. Five injection flow rates were used, which are listed in Table 4.2 along with the corresponding velocities.

Table 4.1. Core samples' permeability and contrast ratio to particle
(Size contrast ratio: particle diameter/porous media diameter)

	Experiment #A	Experiment #B
Permeability, mD	34.62	204
Pore size(diameter), μm	2.43	5.63
Contrast ratio before re-crosslink	12.4	28.6
Contrast ratio after re-crosslink	2.3	5.4

Table 4.2. Injection flow rates and their corresponding velocities

Pump flow rate, ml/min	Velocity, ft/day
0.05	0.47
0.075	0.70
0.1	0.93
0.125	1.16
0.15	1.40

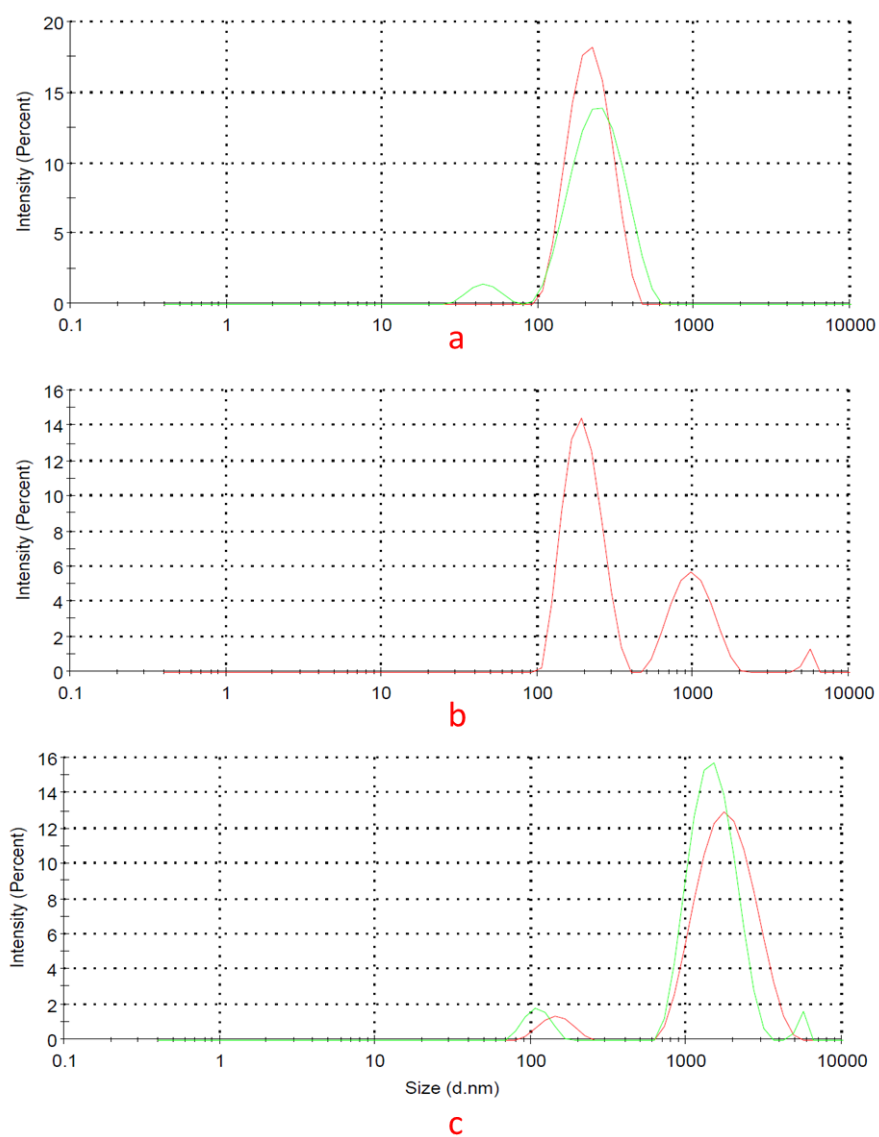


Figure 4.1. Size distributions of Re-crosslinked microgel particles in different conditions
a: before being heated; b: after being heated for one day; c: after being heated for 3 days

Procedures of these two experiments are similar to the procedures in Section 3.1.3, there were two difference:

1. There is no oil saturation and second oil injection steps.

2. After gel injection, core samples would be sealed and placed in an oven with 65°C for three days.

4.1.2.2 The plugging and increasing oil recovery abilities of Re-crosslinked microgel in multiple-phase condition. Oil phase was contained to study the abilities of Re-crosslinked microgel improving oil recovery and plugging in two-phase condition.

In this part, experiments were performed with bigger core samples, which have similar sizes with cores used in previous work which mentioned in Section 3.1.1. Higher injection flow rates were used because of bigger cross-section area. Flow rates are listed in Table 4.3 with corresponding velocities. Four cores with different permeability (252, 102.5, 71.6 and 12.1mD) were selected to study the impact of permeability. Permeability, estimated diameters of porous media and its contrast ratios with particle size are listed in Table 4.4. As shown in Figure 4.2, other than heating core samples with 65°C for three days after gel injection, procedures of these experiments are as same as the procedures listed in Section 3.1.3.

4.1.2.3 The impact of oil phase and oil saturation on microgel transportation. Because of the difference between gel injection in single phase condition and in two-phase condition, additional two core flooding experiments were performed to observe the effect of the oil phase's absence.

The permeability of two core samples used in additional core flooding experiments are similar to the rock permeability of experiment #E. The difference among three experiments are shown in Figure 4.3 by comparing their work flow. The procedures of experiment #G are as same as experiments in Section 4.1.2.1. In experiment #H, after

After gel injection, the resistance factors of experiment #A and #B are 109 and 208 at 0.1 ml/min, respectively. However, during second water flooding, in experiment #A, injection pressure is even higher than gel injection pressure. On the other hand, in experiment #B, the residual resistance factor is only 6.54. Table 4.5 shows the residual resistance factors in experiment #A and #B at different flow rates.

Table 4.3. Injection flow rates and their corresponding velocities

Pump flow rate, ml/min	Velocity, ft/day
0.5	1.2
0.75	1.8
1	2.4
1.25	3.0
1.5	3.6

4.2.2. Experiments with Multiple Phases. When oil phase was contained in this part of work, it only took less than 6 PVs of microgel injection volume to obtain stable injection pressure. In experiments #F, microgel dispersion was very hard to be injected into the core sample with a permeability of 12mD since the injection pressure increase to the pressure limit of experimental instruments in a very short period of time.

Table 4.4. Core samples' permeability and contrast ratio to particle

	Experiment #C	Experiment #D	Experiment #E	Experiment #F
Permeability, mD	252	102.5	71.6	12.1
Pore size, μm	6.04	4.14	3.52	1.55
contrast ratio before re-crosslink	1:30.7	1:21.0	1:17.9	1:7.9
contrast ratio after re-crosslink	1:5.8	1:4.0	1:3.4	1:1.5

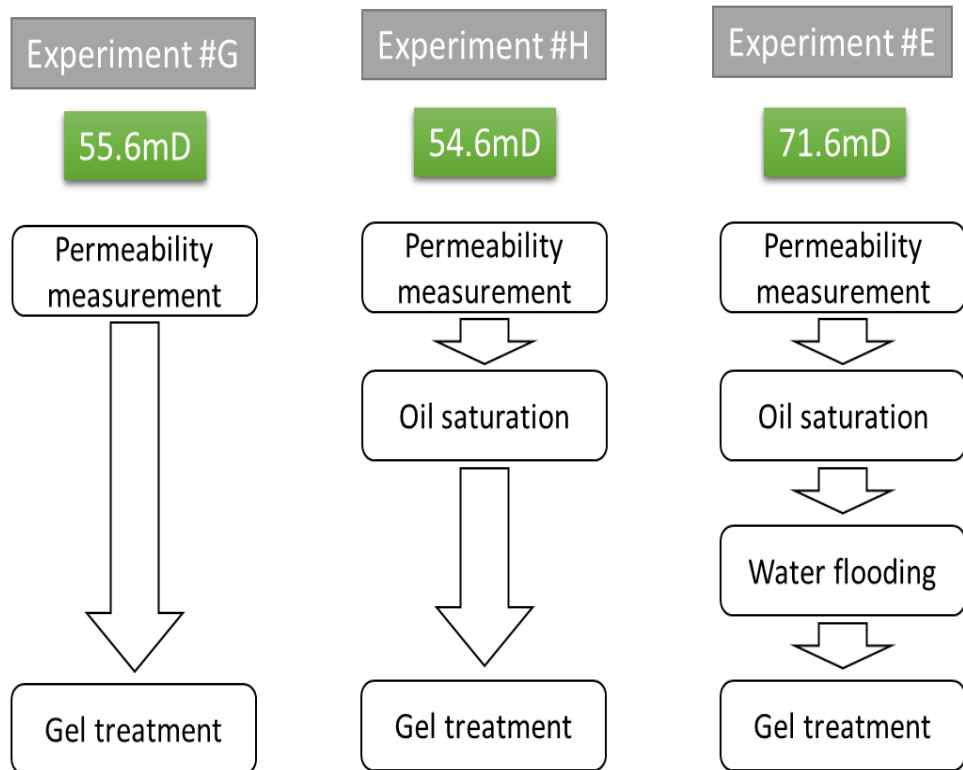


Figure 4.3. Difference among experiments by work flow

Table 4.5. RRFs of experiment #A and #B with different flow rates

Flow rate, ml/min	RRF #A(34.62mD)	RRF #B(204mD)
0.15	119.19	6.1
0.125	129.51	5.83
0.1	150.72	6.54
0.075	174.16	6.45
0.05	184.21	8.14

Figure 4.4 shows gel injection pressure plots with a flow rate of 0.5ml/min. Figure 4.5 shows second water flooding pressure plots at the same flow rate. During gel injection and second water flooding using Re-crosslinked microgel, injection pressure plots had similar trend compare to PAM microgel. Pressure in both steps increased to a peak and then dropped to a stable pressure. Figure 4.6 shows the peaks of resistance factors during gel treatment and the stable resistance factors. Figure 4.7 shows the peaks of residual resistance factors during second water flooding and the residual resistance factors. The flow rate was 0.5ml/min. All the figures show that both resistance factors and residual resistance factors increased with lower permeability.

Figure 4.8, 4.9 and 4.10 show the residual resistance factors to water and oil at varying flow rates in experiment #C, #D and #E. All residual resistance factors to oil are lower than their corresponding residual resistance factors to water, which shows a favorable disproportionate permeability reduction.

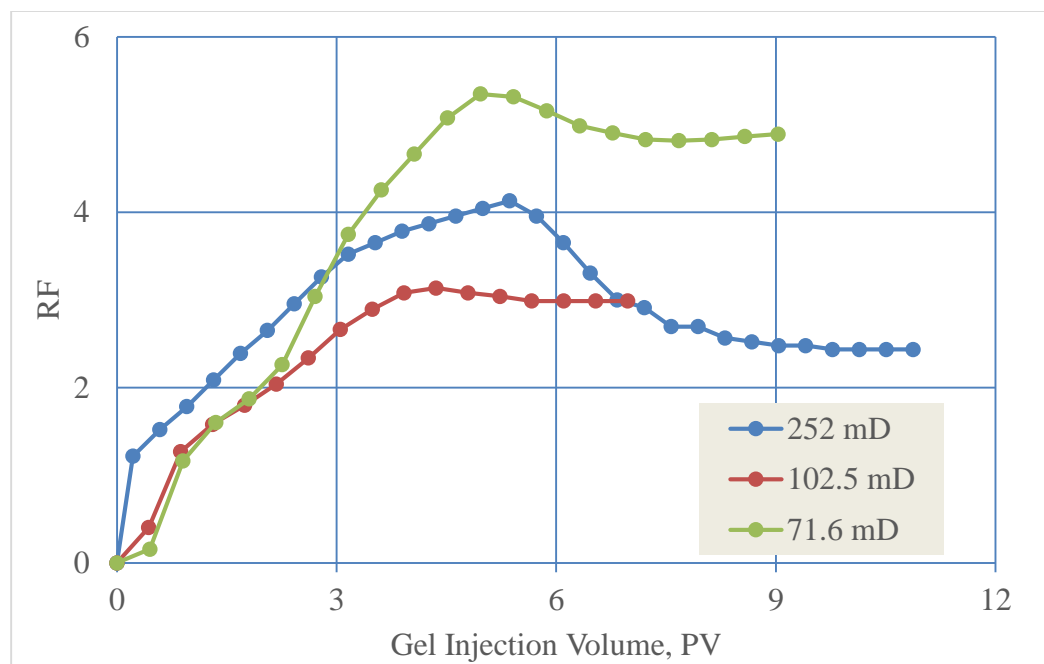


Figure 4.4. Gel injection pressure plots

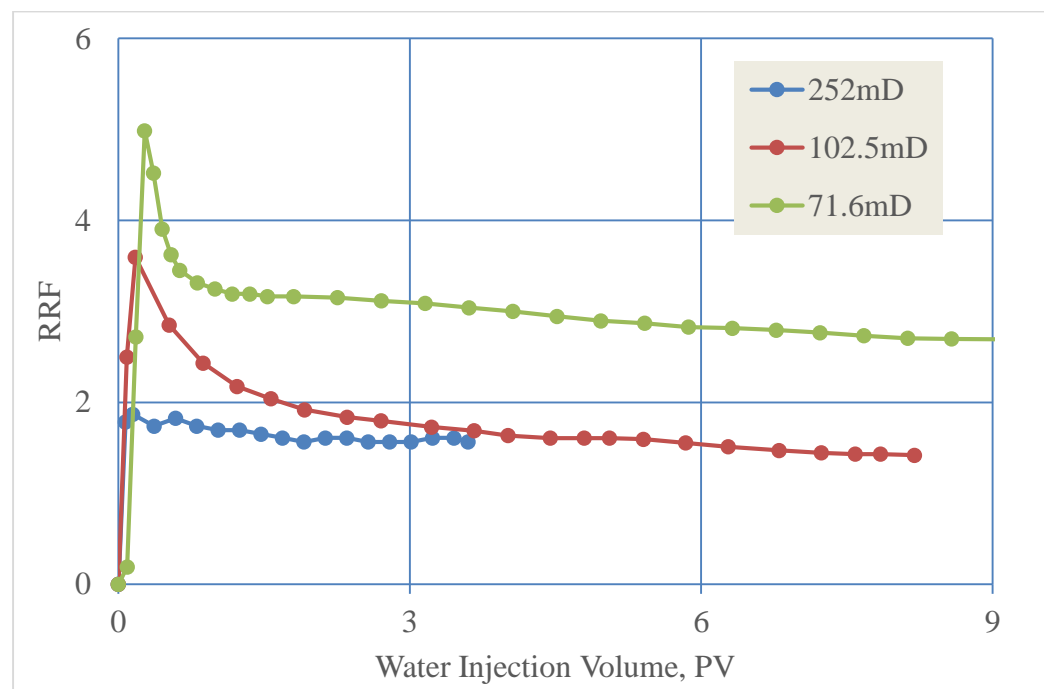


Figure 4.5. Second water flooding pressure plots

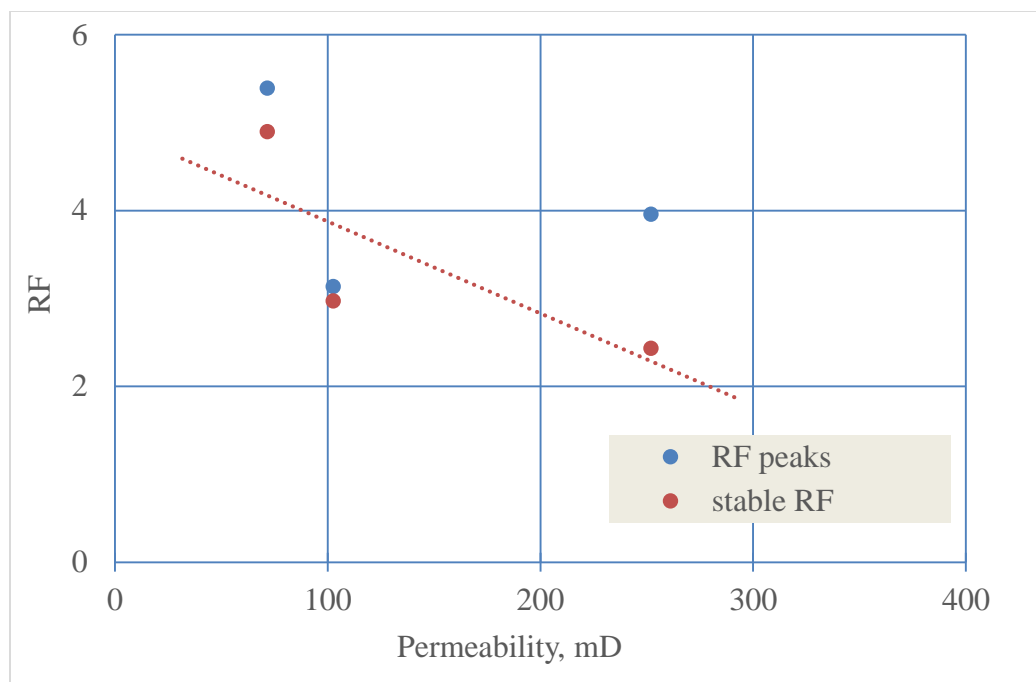


Figure 4.6. Peaks of resistance factor during gel injection and the resistance factor

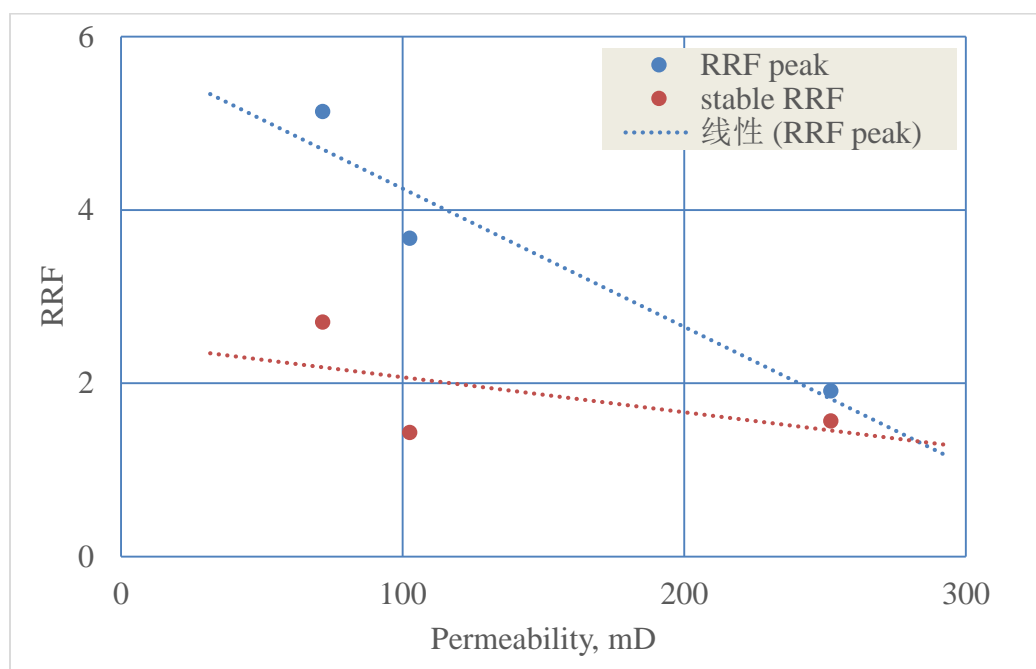


Figure 4.7. The peaks of residual resistance factor and the residual resistance factor

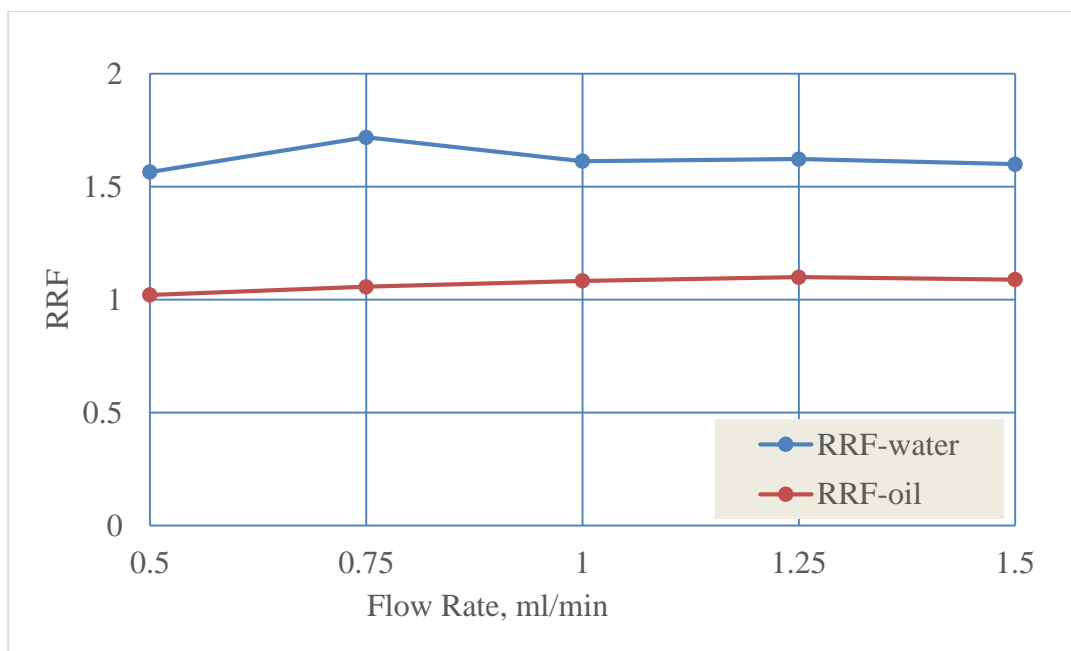


Figure 4.8. Residual resistance factors of water and oil in experiment #C

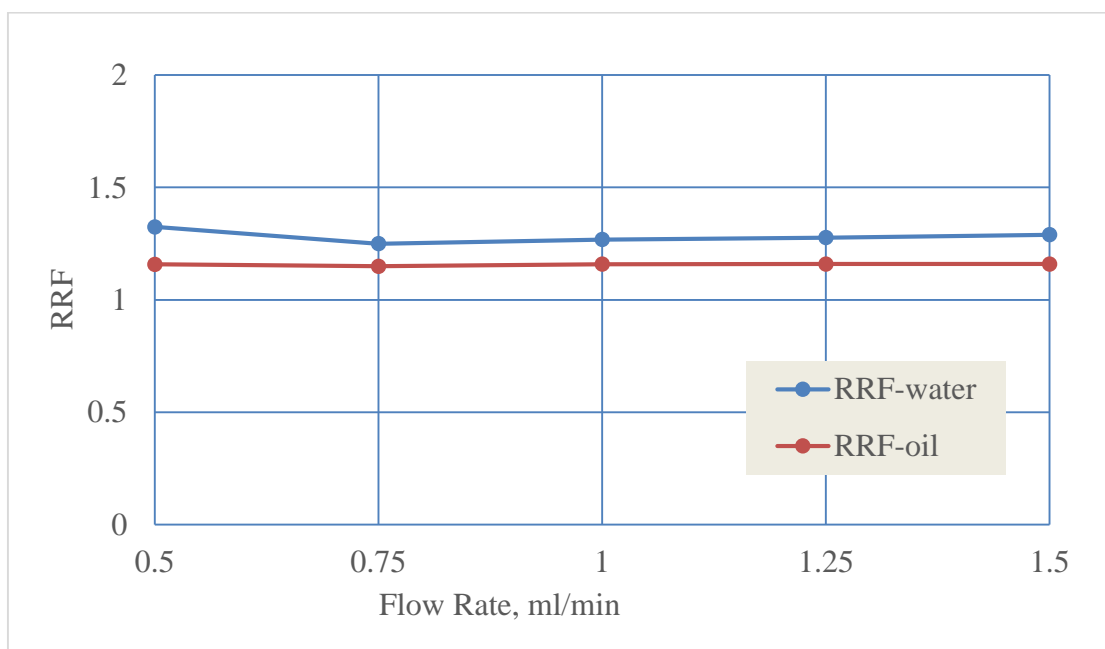


Figure 4.9. Residual resistance factors of water and oil in experiment #D

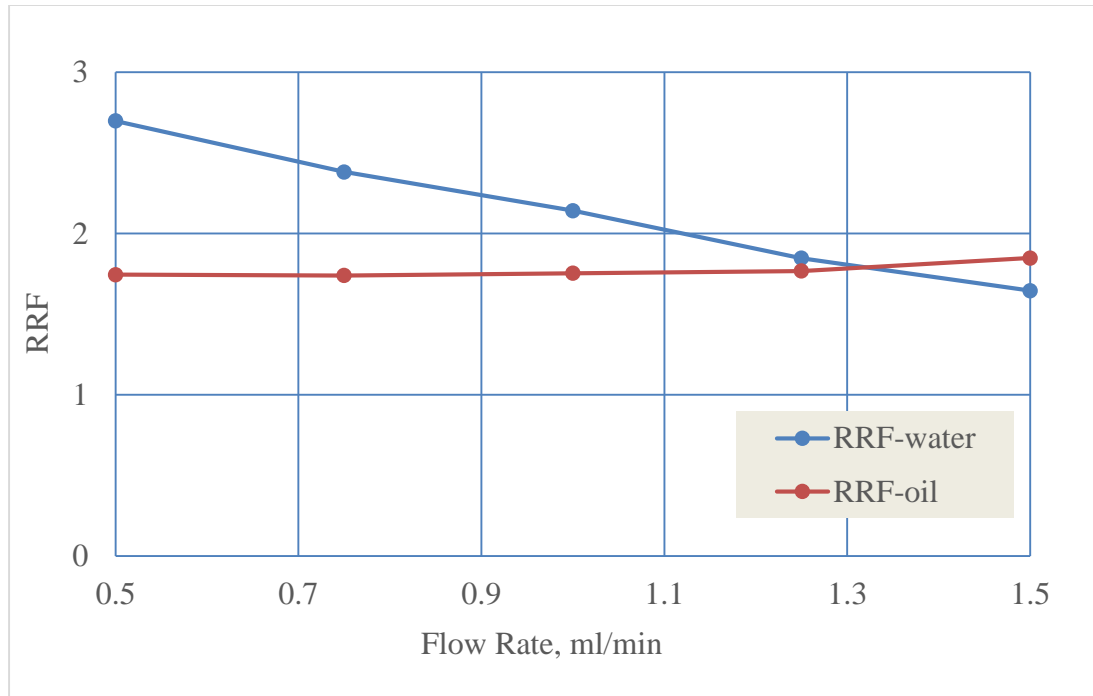


Figure 4.10. Residual resistance factors of water and oil in experiment #E

Table 4.6 shows the oil recovery factors in experiment #C, #D and #E. The total oil recovery increment is higher when permeability is lower. Meanwhile, there were 7.8% oil recovery increment during gel injection in experiment #F, which also fit this trend.

4.2.3. The Effect of Oil Phase on Gel Injection Process. The major difference among three experiments in this part is the oil saturation. At the moment of microgel injection, the oil saturation in experiment #G, #H and #E are 0, 70.36% and 36.84% respectively.

When oil saturation was 0, injection pressure cannot reach peak or stay stable even after an injection volume of 20PVs. The injection pressure plot is shown in Figure 4.11. As shown in Figure 4.12, when oil saturation was 36.84%, injection pressure

reached the maximum value after 2.5PVs of injection volume and then drop to a stable value after total 4PVs of microgel being injected. As shown in Figure 4.13, when the oil

Table 4.6. Oil recovery factors of different stages
(W.F.: water flooding; Gel: gel treatment; 2nd W.F.: second water flooding)

Experiment	Permeability, mD	Oil recovery factors, %			Oil recovery increased, %
		W.F.	Gel	2nd W.F.	
C	252	57.24	0.26	0.39	0.65
D	102.5	43.86	3.73	0.47	4.2
E	71.6	49.03	6.45	0.75	7.2

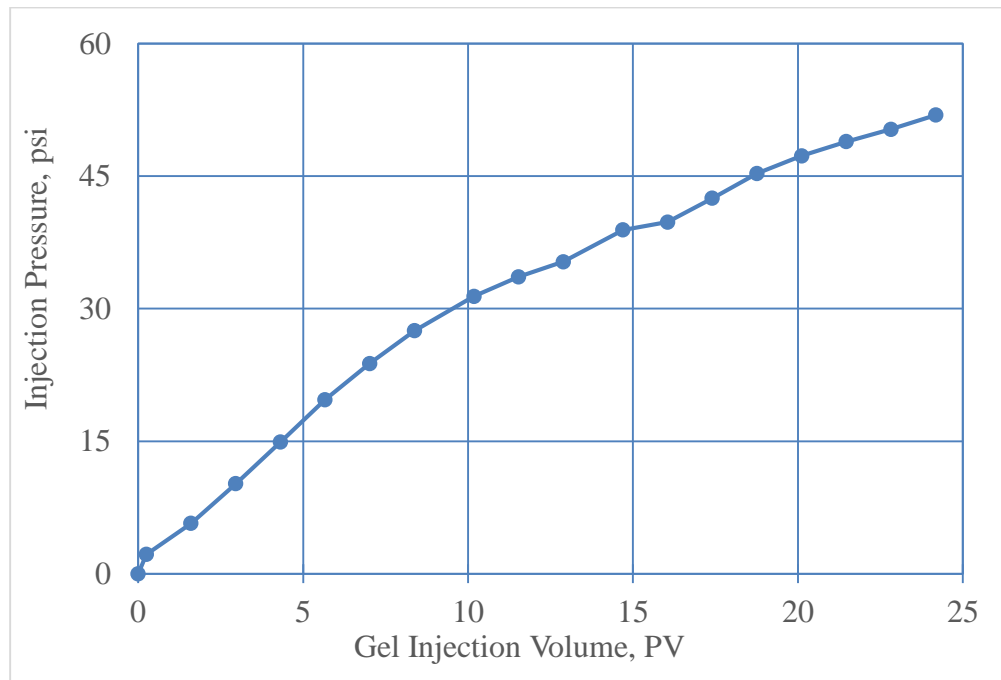


Figure 4.11. Gel injection pressure plot of experiment #G

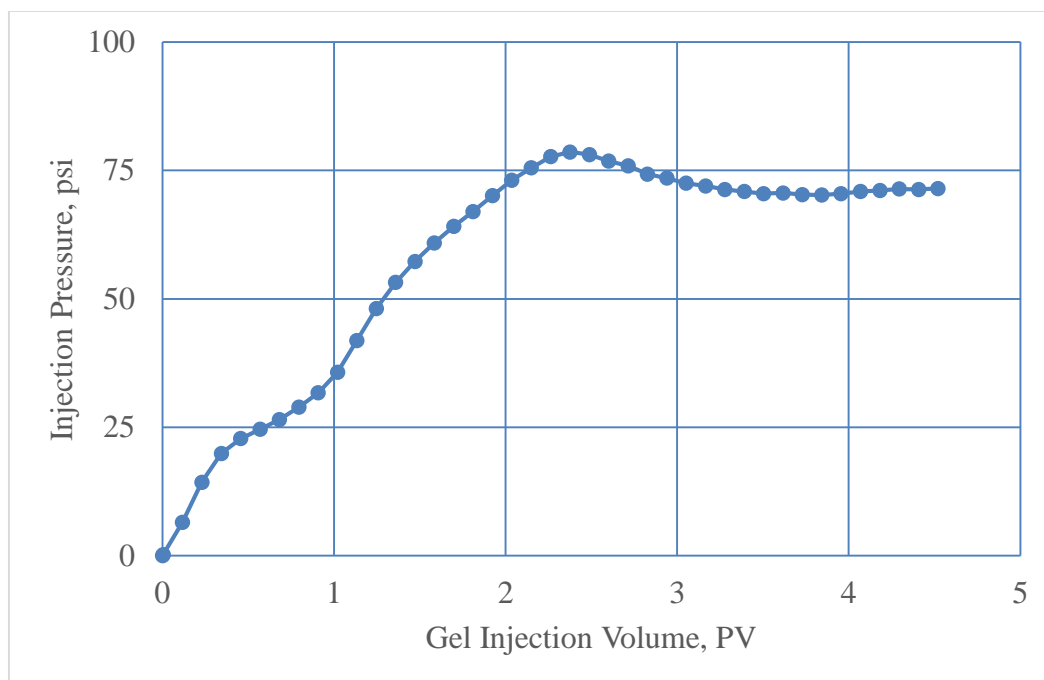


Figure 4.12. Gel injection pressure plot of experiment #H

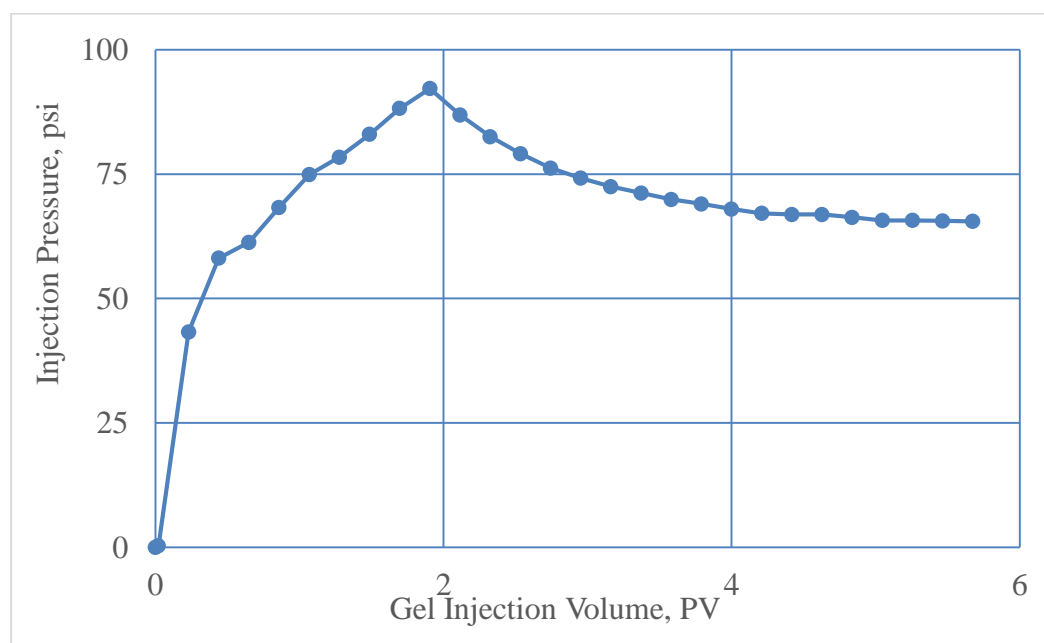


Figure 4.13. Gel injection pressure plot of experiment #E

saturation was 70.36%, only after less than 2PVs of microgel injection volume, the pressure reached the peak and then dropped to a stable value when total 5PVs of gel dispersion being injected.

4.3. DATA ANALYSIS AND DISCUSSION

Experiment #C, #D and #E show that Re-crosslinked microgel have better performance on both oil recovery improvement and plugging aspects with lower permeability. Such result shows re-crosslinked microgel particle is stronger than PAM microgel since the former still had good plug efficiency even with a high injection pressure. Meanwhile, when PAM microgel was being injected, after injection pressures reach the peak, it would drop an average of 38% to a stable value. On the other hand, when Re-crosslinked microgel was being injected, injection pressures only drop an average of 22% to a stable value. Moreover, when the permeability of core sample was 12.1mD, it was extremely hard to inject such microgel into the core sample. However, with the similar particle/pore size contrast ratio applied to PAM microgel, the microgel treatment was easier.

Meanwhile, the results also indicate when there is oil phase involving in experiments, it took much less injection volume to obtain stable injection pressure. The results from experiment #G, #H and #E also show that with higher oil saturation, gel injection process require less microgel amount. In experiment #E, it took more injection volume of microgel to obtain stable injection pressure because the injection flow was transferring from two-phase flow (oil and water phase) to relatively one-phase flow (only water phase).

In both experiment #A and #B, large amount of microgel dispersion was injected into core samples and both resistance factors are higher than 100. However, the residual resistance factors of two experiments shows big diversity as shown in Table 4.5. the plugging efficiency is better with lower rock permeability.

4.4. SUMMARY

Eight core flooding experiments were run to understand the effect of a newly developed re-crosslinked particle gels on plugging and improving oil recovery, and the following conclusions can be drawn:

1. In single-phase condition, it took longer time for the microgel injection to reach a stable injection pressure. Meanwhile, a low permeability core resulted in high residual resistance factor.
2. In water/oil two-phase condition, less microgel is needed to reach stable pressure. Meanwhile, the microgel had a better performance on both plugging efficiency and oil recovery improvement in lower permeability core samples. However, when the core permeability was lower to 12.1 mD, the microgel injection pressure increased sharply to the upper limit of our designed pressure, indicating the microgel cannot propagate in the rocks.
3. Microgel transported faster in higher oil saturation sandstone than low oil saturation sandstone.
4. Re-crosslinked microgel has a better strength and DPR comparing to PAM microgel.

5. CONCLUSION

This research evaluated two types of microgels (PAM microgel and Re-crosslinked microgel) designed for conformance control by core flooding experiments. Both microgels were synthesized in our lab. Following conclusions could be drawn from this work.

1. Six core flooding experiments were performed to understand the effect of crosslinker concentration and permeability on PAM microgel plugging and improving oil recovery in porous media. Results show that:
 - a. PAM microgel synthesized by lower concentration of crosslinker has higher swelling ratio.
 - b. For the microgel synthesized by same crosslinker concentration, microgel treatment would result in low plugging efficiency in lower permeability rocks because of the poor strength under high injection pressure.
 - c. Within a same permeability range of cores, the microgel synthesized by lower crosslinker concentration will have better plugging efficiency.
2. Eight core flooding experiments using Re-crosslinked microgel were performed to study the impact of oil saturation and permeability on gel treatment, following conclusions could be drawn:
 - a. It took much longer time for microgel injection to reach stable pressure in water saturated rocks than in the rocks with oil.
 - b. The residual resistance factors are lower in higher permeability sandstones when the rocks were only saturated with water.

- c. In water/oil two-phase condition, microgel had better plugging efficiency in lower permeability porous media.
 - d. Microgel had better transportation ability in the rocks with higher oil saturation.
3. Comparing both microgels by the experimental results, the following conclusions could be drawn:
- a. A smaller particle/pore size contrast ratio, which means bigger particle size or lower permeability, is favorable for improving oil recovery.
 - b. Re-crosslinked microgel had better plugging efficiency than PAM microgel.
 - c. Re-crosslinked microgel showed a more favorable disproportionate permeability reduction effect comparing PAM microgel.

6. FUTURE WORKS

The core flooding results showed that microgels can improve the oil recovery in homogenous cores during microgel treatment and following water flooding process. However, the mechanisms behind it are not understood yet, which need further study by evaluating the interaction of microgels with fluids and rock surface. In addition, microgel treatment mainly target on heterogeneous reservoirs; therefore, more experiments are needed to be performed using heterogeneous models to evaluate sweep efficiency improvement by microgels.

Experimental results show that the transportation of microgels through porous media is strongly affected by oil saturation; however, the mechanism behind this result is not clear to us either. More experiments need to be carried out to quantify the effect of different parameters on microgel transportation and retention.

BIBLIOGRAPHY

1. Green, D.W. and G.P. Willhite, Enhanced Oil Recovery. 1997, Richardson, US: Society of Petroleum Engineers.
2. Terry, R.E., Enhanced oil recovery. Encyclopedia of physical science and technology, 2001. **18**: p. 503-518.
3. Alvarado, V. and E. Manrique, Enhanced Oil Recovery - Field Planning and Development Strategies. 2010, Elsevier.
4. Bailey, B., et al., Water control. Oilfield Review, 2000. **12**(1): p. 30-51.
5. Sorbie, K.S. and R.S. Seright, Gel Placement in Heterogeneous Systems With Crossflow. 1992, Society of Petroleum Engineers.
6. McLaughlin, H.C., J. Diller, and H.J. Ayres, Treatment of Injection and Producing Wells with Monomer Solution. 1975, Society of Petroleum Engineers.
7. Norman, C.A., J.E. Smith, and R.S. Thompson, Economics of In-Depth Polymer Gel Processes. 1999, Society of Petroleum Engineers.
8. Lantz, M. and G. Muniz, Conformance Improvement Using Polymer Gels: A Case Study Approach. 2014, Society of Petroleum Engineers.
9. Bai, B., et al., Case Study on Prefromed Particle Gel for In-Depth Fluid Diversion. 2008, Society of Petroleum Engineers.
10. Bai, B., M. Wei, and Y. Liu, Field and Lab Experience With a Successful Preformed Particle Gel Conformance Control Technology. 2013, Society of Petroleum Engineers.
11. Chauveteau, G., et al., New Size-Controlled Microgels for Oil Production. 2001, Society of Petroleum Engineers.
12. Feng, Y., et al., Characteristics of Microgels Designed for Water Shutoff and Profile Control. 2003, Society of Petroleum Engineers.
13. Zaitoun, A., et al., Using Microgels to Shut Off Water in a Gas Storage Well. 2007, Society of Petroleum Engineers.
14. Roussennac, B.D. and C. Toschi, Brightwater trial in Salema Field (Campos Basin, Brazil). 2010, Society of Petroleum Engineers.

15. Pritchett, J., et al., Field Application of a New In-Depth Waterflood Conformance Improvement Tool. 2003, Society of Petroleum Engineers.
16. Ohms, D., et al., Incremental-Oil Success From Waterflood Sweep Improvement in Alaska. 2010.
17. Askarinezhad, R., D.G. Hatzignatiou, and A. Stavland, Disproportionate Permeability Reduction of Water-Soluble Silicate Gelants - Importance of Formation Wettability. 2016, Society of Petroleum Engineers.
18. Sydansk, R.D. and R.S. Seright, When and Where Relative Permeability Modification Water-Shutoff Treatments Can Be Successfully Applied. 2006, Society of Petroleum Engineers.
19. Liang, J.-T., H. Sun, and R.S. Seright, Why Do Gels Reduce Water Permeability More Than Oil Permeability? 1995.
20. Almohsin, A.M., et al., Transport of Nanogel through Porous Media and Its Resistance to Water Flow. 2014, Society of Petroleum Engineers.
21. Goudarzi, A., et al., New Experiments and Models for Conformance Control Microgels. 2014, Society of Petroleum Engineers.
22. Dupuis, G., et al., Successful Chemical Water Shut-Off Treatment in an Omani Field Heavy-Oil Well. 2015. Society of Petroleum Engineers.
23. Dupuis, G., et al., Water Conformance Treatment using SMG Microgels: A Successful Field Case. 2016, Society of Petroleum Engineers.
24. Salehi, M., et al., Performance Evaluation of Thermally-Activated Polymers for Conformance Correction Applications. 2012, Society of Petroleum Engineers.
25. Fabbri, C., et al., Laboratory and Simulation Investigation of the Effect of Thermally Activated Polymer on Permeability Reduction in Highly Permeable Unconsolidated Sand. 2015, Society of Petroleum Engineers.
26. Chuan-jin, Y., et al., Study on Plugging Performance of Pore-scale Elastic Microspheres. Science Technology and Engineering, 2012(06): p. 1244-1247.
27. Guanglun, L., et al., Study of influencing factors on pore-scale elastic microspheres flooding. Petroleum Geology and Recovery Efficiency, 2012(02): p. 41-43+113-114.
28. Dai-Feng, S., et al., Further Enhanced Oil Recovery by Using PolymerMinispheres at GudaoOil Field after Polymer Flood. Oilfield Chemistry, 2008(02): p. 165-169+185.

29. Li-mei, Z., Application of Alkali Resistant Polymer Microsphere Grain Profile Technology in ASP. Science Technology and Engineering, 2012(22): p. 5602-5605.
30. Zhang, Y., et al., Controllable Preparation and Properties of Large Size Polymer Micro. POLYMER MATERIALS SCIENCE AND ENGINEERING, 2013(05): p. 169-172+176.
31. Lin, W., et al., Evaluation and application of YG polymer microspheres as deep profile control agent. Oilfield Chemistry, 2011(03): p. 327-330.
32. Jiao, L., et al., PREPARATION AND PLUGGING PROPERTIES OF PRE-CROSSLINKED POLYACRYLAMIDE DISPERSION. ACTA PETROLEI SINICA (PETROLEUM PROCESSING SECTION), 2010(05): p. 800-805.
33. Imqam, A., B. Bai, and M. Wei, Combining Conformance Treatment with Mobility Control Improves Oil Sweep Efficiency in Non-Cross Flow Heterogeneous Reservoirs. 2015, Society of Petroleum Engineers.
34. Cleveland-Quarries, Berea Sandstone™ Petroleum Cores.
<http://www.bereasandstonecores.com/>.

VITA

Haifeng Ding received his Bachelor of Science degree in petroleum engineering from China University of Petroleum-Beijing in June 2014. He started to pursue his master's degree in petroleum engineering of Missouri University of Science and Technology in August, 2014. He joined Dr. Baojun Bai's research group in September, 2014 and worked as a research assistant. In December, 2016, he received his Master of Science in Petroleum Engineering from Missouri University of Science and Technology.