

Viscous Effects of Wetting and Non-Wetting Fluids on
Capillary Trapping Mechanism: A Climate Change Mitigation Strategy.

by

Simone Fobi

A PROJECT

Submitted to

Oregon State University

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degree of

Honors Baccalaureate of Science in Environmental Engineering (Honors Associate)

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Dorthe Wildenschild

Rising CO₂ concentrations in the atmosphere (396 ppmv as of April 2012) increase the effect of global warming and climate change. CO₂ sequestration has become a potential method to mitigate climate change. This study focuses on capillary/residual trapping as a form of geologic CO₂ sequestration. Capillary/residual trapping occurs when supercritical CO₂ is stored in rock pore spaces after injection at high temperatures and pressures. This project aims to isolate the effects of viscosity from that of interfacial tension, which also influences capillary/residual trapping. Understanding how viscosity affects capillary/residual trapping is a key to determine optimal injection temperatures and pressures, which maximize CO₂ trapping. Two viscothickeners, guar gum and glycerol were used to vary the viscosity of KI brine without changing interfacial tension. Subsequently, guar gum and KI brine mixtures were injected into rock cores to determine capillary/residual potential. Results suggest that higher viscosities increase residual trapping potential. This study provides an initial analysis of the role of viscous effects in capillary trapping of CO₂.

Key Words: Viscothickener, CO₂ sequestration, capillary/residual trapping, surface tension, guar gum.

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presented on June 1st 2012.

APPROVED:

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I understand that my project will become part of the permanent collection of Oregon State University, University Honors College. My signature below authorizes release of my project to any reader upon request.

Simone Fobi, Author

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CHAPTER I: SCOPE OF STUDY

The thesis objective is to investigate the feasibility of CO₂ storage in saline formations, as a climate change mitigation strategy. According to many scientists such as Howard Herzog (Principal Research Engineer at Massachusetts Institute of Technology), hundreds to thousands GtC of carbon can be stored in deep saline formations [17]. This thesis focuses specifically on capillary trapping as the key storage mechanism within these saline formations. The goal is to evaluate capillary trapping efficiency as a function of viscosity. This thesis aims to isolate viscous effects from that of other factors such as surface tension, which also influence capillary trapping.

A literature review is presented as a basis for understanding CO₂ sequestration, trapping mechanisms in geologic formations, factors affecting trapping of CO₂ and methods for changing viscosity. Methods and results from lab-scale experiments will also be covered in this thesis. Recommendations and future work will be proposed.

This thesis was partly sponsored by the Wildenschild Laboratory at Oregon State University, with the guidance of Dr. Dorte Wildenschild. It was also sponsored by Oregon State University Honors College and was written to meet the Oregon State University Honors College requirements. This thesis would not be possible without tremendous support from both sponsors.

CHAPTER II: LITERATURE REVIEW

2.0 Introduction

Over the past decade, global warming and ocean acidification have been on the rise, as greenhouse gas concentrations in the atmosphere have greatly increased. The presence of these greenhouse gases has been linked to rising temperatures and extreme weather events such as hurricanes and floods. The concentration of gases like CO₂ and CH₄ in the atmosphere has been exacerbated due to anthropogenic activities, where current CO₂ concentrations as of April 2012 are 396 ppmv compared to 280 ppmv in Pre-industrial times [1]. It is of importance to mitigate the presence of such gases in the atmosphere. The objective of this literature review is to understand climate mitigation strategies through CO₂ sequestration. An overview of sequestration techniques, the key factors in CO₂ sequestration and a proposed approach to CO₂ storage will be explored in this section.

2.1 CO₂ sequestration

CO₂ sequestration is the process by which CO₂ is removed from the atmosphere. Natural or engineered physical, biological and chemical processes can be used to reduce CO₂ concentrations in the atmosphere. CO₂ sequestration is a recognized approach to removing CO₂ from the environment and thus mitigates climate impacts.

Geological sequestration is the physical injection of CO₂ into geologic formations where porous rock layers serve as a storage reservoir. Currently less than a tenth of a thousand of globally produced anthropogenic CO₂ is stored geologically [2]. This shows that there is room for growth in this sector. Ocean storage, similar to geologic storage

consists of the injection of CO₂ in oceans at depths greater than 1000 m [2]. In this case CO₂ dissolves and becomes part of the carbon cycle.

Chemical sequestration is characterized mainly by mineral carbonation. Minerals like calcite and magnesite are precipitated out of solution upon reaction with CO₂, forming silicates rich in calcium and magnesium. The advantage of chemical sequestration is that CO₂ stored in this form is stable over long time periods. Resulting constituents can be used to reinforce concrete or as an amendment to soil.

Biological sequestration occurs primary through storage in soil and biomass. Forests and soils are the primary sinks for CO₂ in biological sequestration. Biological sequestration through forestation is rendered less effective because, plants also give off CO₂ during their nighttime respiration. Success of biological sequestration depends on management of forests and soil to ensure that CO₂ is not returned to the atmosphere, either through burning biomass or soil tillage [3].

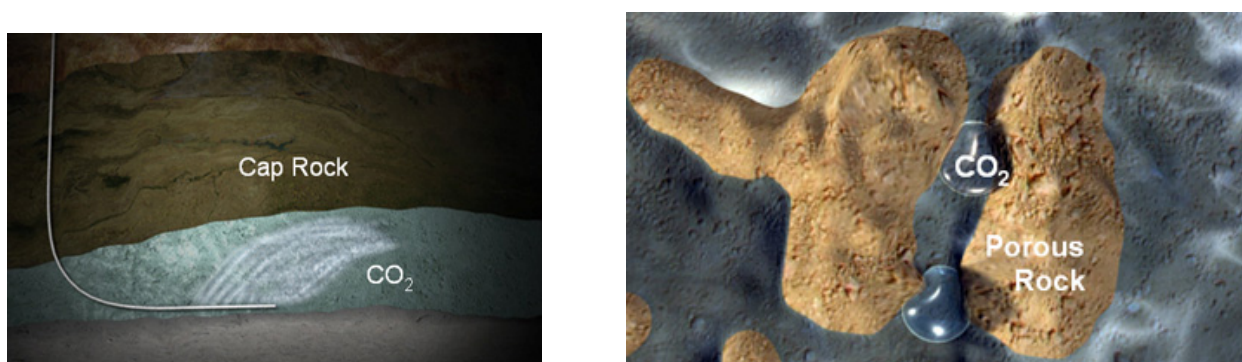
Geologic sequestration is known to have the largest CO₂ storage potential, significant enough to result in a noticeable reduction in atmospheric CO₂, on the order of a billion metric tons or more could be stored in these formations annually [4]. Thus geologic sequestration will be of focus throughout this thesis.

2.2 Types of geologic sequestration

Geologic CO₂ sequestration is the storage of CO₂ in porous rock formations, which could be deep oil reservoirs or sedimentary basins [2]. Sedimentary basins are thought best to capture CO₂ due to their high pore volume and connectivity [4]. Injection

of CO₂ into these geologic formations can capture CO₂ by four main processes: structural, solubility, capillary/residual and mineral trapping.

Structural Trapping, the most dominant trapping mechanism occurs when an impermeable cap rock, impedes the upward movement of CO₂ after injection into the ground. **Figure 1a** shows structural trapping of CO₂ by an impermeable cap rock. The fluid is held in a dome of the structure [5]. A potential problem with structural trapping is potential leakage from the rock, if fractures are present.



From: http://www.co2captureproject.org/co2_trapping.html [16].

(a)

(b)

Figure 1: a) Structural trapping of supercritical CO₂ by an impermeable cap rock after injection of CO₂ in saline geologic formation. b) Capillary/Residual trapping of supercritical CO₂ where CO₂ bubbles are held in between grains in the porous spaces of rock. Both trapping mechanisms are means to sequester CO₂ in geologic formations [6].

During injection of CO₂, saline solution is displaced and this process is known as drainage. After injection, the brine refills the pore spaces occupied by CO₂ and this process is referred to as imbibition. During imbibition, tiny CO₂ bubbles are trapped within the pore spaces of the rocks and are held in place mainly by capillary forces. This process is known as *Capillary/Residual Trapping*. **Figure 1b** depicts capillary/residual trapping of CO₂ in porous rocks [6].

After injection, some of the CO_2 dissolves in brine solution and this process is known as *Solubility Trapping*. Dissolution of CO_2 into saline solution creates a denser fluid, which may then sink to the bottom of the formation. Over extended periods of time, chemical reactions of dissolved CO_2 may form minerals, which will then precipitate from solution. Precipitation of these minerals from solution is known as *Mineral Trapping* and it is presumed to be the most secure type of trapping [5].

Structural trapping occurs on shorter time scales while mineral trapping happens on long time scales of thousands of years. **Figure 2** shows each trapping mechanism as a function of time. Mineral trapping is the most secure form of CO_2 trapping with little to no leakage or remobilization potential.

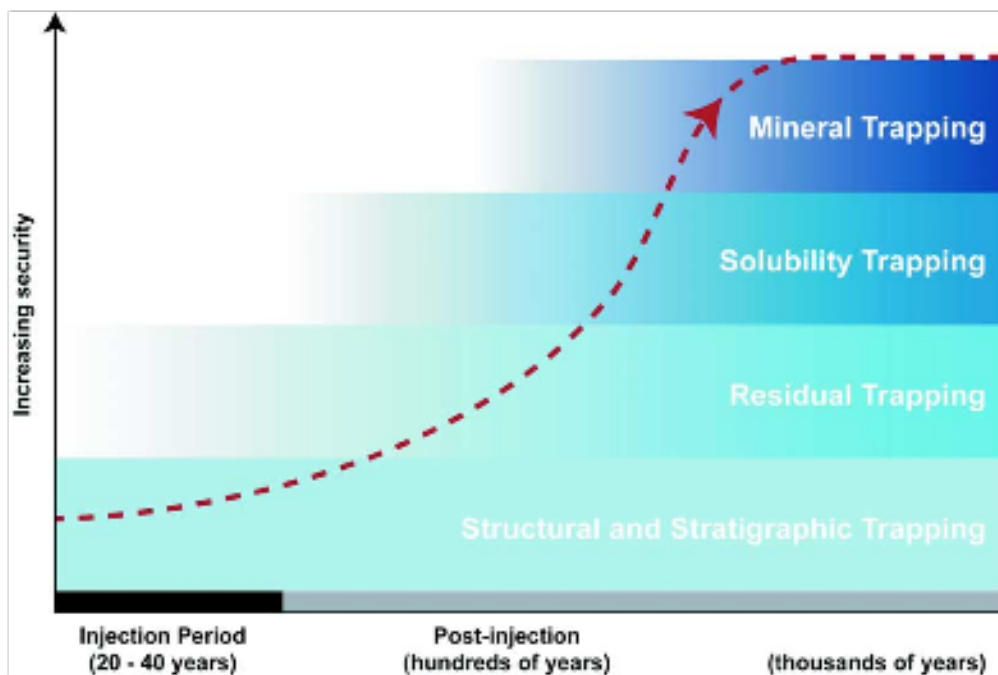


Figure 2: Types of geologic CO_2 sequestration as a function of time and trapping security. Structural trapping offers the shortest time scale with the least storage security while mineral trapping occurs on long time scales but has the highest storage security [5].

Residual trapping is advantageous because it also promotes dissolution of CO_2 in saline solution. Bubble formations provides large surface to volume ratios, which

enhance dissolution. The increased storage capacity associated with capillary/residual trapping makes it favorable for further investigation [6]. The mechanism is explained in more detail in the following section.

2.3 Residual or capillary trapping

CO₂ is typically injected at depths greater than 800 m, at elevated temperatures and pressures. In these regions, CO₂ exist in its supercritical state (temperatures ≥ 30 °C and pressures ≥ 7 MPa [7]) and it occupies less volume because it is denser than CO₂ at ambient conditions. Supercritical CO₂ is temperature and pressure dependent and these in turn affect the viscosity of the injected fluid as shown in **Figure 3**. Understanding how viscosity controls capillary/residual trapping, we can determine optimal injection temperatures and pressures for supercritical CO₂. It is important to note that the viscosities shown in this plot are for CO₂ and therefore the non-wetting fluid in the system.

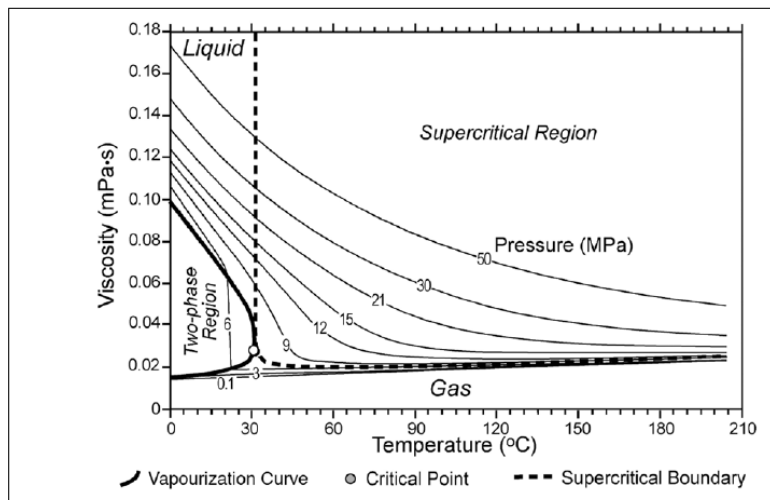


Figure A1.4 Variation of CO₂ viscosity as a function of temperature and pressure (Bachu, 2003).

Figure 3: Viscosity of supercritical CO₂ as a function of temperature and pressure. This plot shows that optimal viscosities can be used to determine best temperature and pressure for CO₂ injection [8].

In order to understand capillary/residual trapping in subsurface formations, it is necessary to consider wettability of the rock. Wettability can be defined as the preference for one fluid to remain in contact with the host solid phase. **Figure 4** shows the interaction between wetting and non-wetting fluids in a model system (a capillary tube), which influences the capillary pressure between the fluid phases.

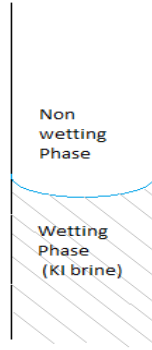


Figure 4: Interaction of wetting and non-wetting fluids, where CO_2 is the non-wetting fluid in natural rock systems and KI brine is the wetting fluid.

In natural rocks, supercritical CO_2 acts as the non-wetting phase while saline solutions act as the wetting phase.

Capillary trapping is controlled by the interactions of viscous and capillary forces. We define capillary number as the ratio of viscous forces to interfacial forces. The relationship is shown in **Equation 1**, where μ_w is wetting phase viscosity, v_w is Darcy fluid velocity for the wetting phase and σ is interfacial tension [9].

$$Ca\# = \frac{\mu_w v_w}{\sigma} \quad (1)$$

The capillary number (Ca) controls whether CO_2 will be trapped or mobilized in pores. Changes to any of the above parameters can show the effects of each parameter on

capillary number and by consequence residual (trapped) CO₂ saturation. A benchmark for this study is also to understand how changes to viscosity affect the capillary number once the solution is injected into rock pores.

2.4 Viscosity study

In order to isolate and understand viscous effects on capillary trapping, attempts to change viscosity of the wetting fluid (KI brine) and the non-wetting fluid (CO₂ proxies) were made. Of particular interest is the alteration of viscosity without changing interfacial tension such that viscous effects can be separated from interfacial effects. This section outlines the viscothickeners used for the wetting and non-wetting fluid.

2.4.1 Viscothickeners for wetting fluid

Guar gum and glycerol were used to change viscosity of the wetting fluid (KI brine). Both viscothickeners were mixed with KI brine and the viscosities were measured.

Guar gum is a polysaccharide compound made mainly of a mannose backbone and galactose side chains. The powdered form of guar gum is obtained from processing the seeds of the compound [10]. Guar gum is popular for its rheological properties as highly viscous solutions can be attained when it is mixed in water. Guar gum hydrates better in water than in organics solvents. Due to its non-Newtonian behavior its viscosity decreases with increases in shear stress. Increasing temperature also decreases the viscosity of guar gum while increasing salinity has been reported to increase the viscosity of the solution [11]. Guar gum is usually sold in its powdered form as a result, it is rare to have reported viscosities for highly concentrated guar gum solutions.

Glycerol or glycerin was also investigated in this study. Glycerol is a polyol compound with hydroxyl groups, which make it water soluble. Glycerol is a colorless, odorless solution. Pure glycerol has a viscosity of 1410 cP and a surface tension with air of 63.5 dynes/cm [12]. **Figure 5** shows the structural layout of both guar gum and glycerol.

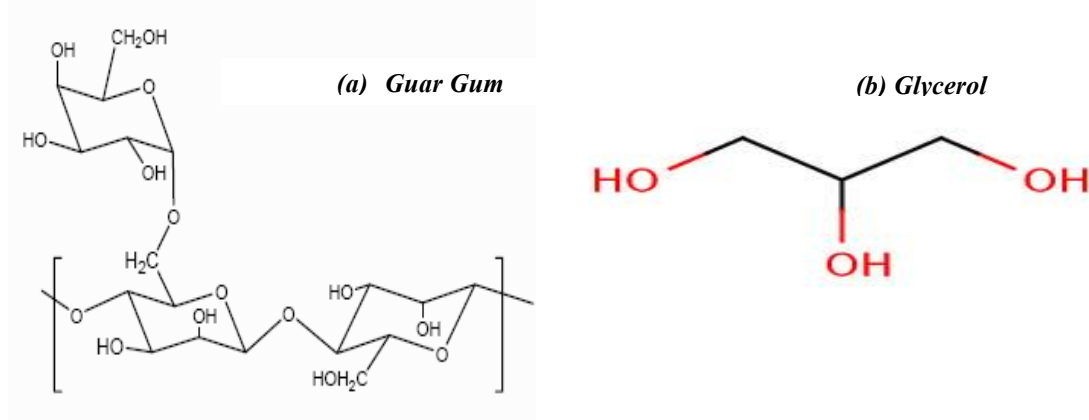


Figure 5: Molecular structures for (a) guar gum and (b) glycerol, respectively. Guar gum consists of large chain hydrocarbons. The hydroxyl groups on both molecules make them water soluble [10].

2.4.2 Viscothickeners for non-wetting fluid (CO₂ proxy fluids)

Attempts to change viscosity of the non-wetting phase were made using silicon oils. Polydimethylsiloxane (PDMS) structure contains siloxane polymers and the solution is a clear and nontoxic oil. It is insoluble in water and ethanol but remains soluble in other oils [13]. Preliminary experiments with PDMS showed inconclusive results as the surface tension varied largely with different viscosities.

Hatzikiriakos and Ochoa [14] suggest another method by which to change the viscosity of non-wetting fluids. Based on their research, Dioctyl Sulfosuccinate Sodium salt (AOT) at different weight fractions was used to increase the viscosity of ISOPAR G

(naphtha solvent) [14]. Their obtained results showed minimal changes in surface tension, as shown in **Table 1**.

Table 1 shows results obtained by Hatzikiriakos and Ochoa [14] when AOT was used to change the viscosity of ISOPAR G. Surface tensions remained fairly constant. Literature values shown below suggest that ISOPAR could be used as a CO₂ proxy fluid and its viscosity can be changed with AOT.

AOT wt % concentration	Surface tension (dynes/cm)	Viscosity (cP)
0	23.5	1
10.03	23.6	1.22
20.22	23.6	1.67
30.26	23.3	2.33
39.41	23.4	4.48
56.5	23.9	10.05

Results obtained from this paper suggest that ISOPAR G could be used as a CO₂ proxy fluid if AOT can increase its viscosity without significantly changing the surface tension. AOT could therefore be used as a viscothickener for the non-wetting phase.

CHAPTER II: METHODS & MATERIALS

3.1 Changing viscosity of the wetting fluid

Viscosity of the wetting fluid (KI brine) was changed using guar gum and glycerol. This section outlines methods used to prepare solutions which were then used during subsequent experiments.

3.1.1 Guar gum dissolution in KI brine

Powdered guar gum (CAS-No: 9000-30-0) was obtained from Sigma Aldrich. Potassium Iodide (KI) was mixed with DI-water in a 1:6 mass ratio respectively, to form the KI brine solution. A Proctor Silex (125 watt) home blender was used to mix guar gum with KI brine in a 1:100 mass ratio. The solution was mixed for 30 – 40 minutes at level 5 speed, typically until a thick slurry formed. Due to air bubble formation in the solution, the slurry was covered with cellophane and left to rest for 3 to 4 hours until most of the bubbles escaped the solution. The solution was poured into eight 45 mL centrifuge tubes and it was centrifuged. The centrifuge was set to 9000 rpms and the solutions were centrifuged for 15 minutes to separate the undissolved guar gum from the liquid phase. The liquid guar gum solutions were then poured into a 500 mL Erlenmeyer flask and used for later experiments.

3.1.2 Glycerol dissolution in KI brine

Potassium Iodide solution was also mixed in a 1:6 mass ratio with water. The formed brine was used to dilute Glycerol (CAS-No:56-81-5) which was purchased from OSU Chemical Store.

3.1.3 Mixing ratios, viscosity and surface tension measurements

KI brine was mixed with guar gum and glycerol to alter the viscosity of the wetting fluid. Volumetric mixing ratios were used to dilute the viscothickener solutions in order to obtain varying dynamic viscosities. Mixing ratios in this study were defined as the volume of viscothickener to the volume of KI brine used. Mixing ratios of dissolved guar gum to KI brine ranged from 0.05 to 0.33. Mixing ratios of glycerol to KI brine ranged from 0.1 to 1. Solutions of varying viscosities were obtained using this method.

Viscosity of prepared solutions was measured using a Cannon Ubbelohde Size 75 viscometer. Solutions obtained through the mixing ratios were individually poured into the viscometer. Pure DI-water was used to calibrate the viscometer. The solution was placed in a 20 °C water bath and left to equilibrate for 10 minutes. A level was used to ensure the viscometer was as upright as possible. The temperature of the water bath was recorded using a glass thermometer. A stopwatch was used to record the time it took the fluid to travel between the two marked points on the viscometer. The recorded time was multiplied by the viscometer constant ($K = 0.008$) to obtain a kinematic viscosity. Recorded densities of each prepared solution were used to obtain dynamic viscosities. Triplicates were taken for each sample.

Surface tensions of the created solutions were measured using a Fisher Surface Tensiometer Model 20 and a DuNuoy ring with a mean circumference of 6 cm and an R/r (radius of ring/ radius of ring wire) ratio of 55. Apparent surface tensions were obtained as direct readings from the equipment. Direct readings are considered apparent because the effects of atmospheric pressure acting on top of the ring are not accounted for in the

reading. A correction factor formula, supplied by the tensiometer manufacturer was used to obtain actual surface tensions. The equipment was calibrated with KI brine and triplicates of each sample were taken. **Equation 2** shows the formula used to determine the correction factor (F).

$$F = 0.7250 + \sqrt{\frac{0.01452P}{C^2D} + 0.04534} - \frac{1.679r}{R} \quad (2)$$

Where P is the surface tension from the dial reading, C is the ring circumference, D is density of KI-Brine mixed with the viscothickener, r and R are the radius of the ring wire and the ring, respectively. Density of each sample was determined by measuring the mass of 25 mL of each solution.

3.2 Injection into rock core to measure trapping potential

A consolidated rock core was saturated with CO₂ by flushing the rock with CO₂. A Precision Syringe Pump was used to inject solutions of guar gum and KI brine or glycerol and KI brine into the rock. A 2 cP solution of guar gum and KI brine was used to initially saturate the rock with the wetting fluid. Scans of the rock core were acquired using the OSU ME Micro CT scanner. Initial saturation of the wetting phase was determined through this scan. Drainage of the rock was initiated after scanning of initial saturation. After drainage the rock was left to equilibrate for 2 hours or more until the capillary pressure returned to about zero. Scans of the rock core were also acquired after drainage and the rock core was then imbibed with more guar gum and KI brine solution. A last scan of the rock was taken to determine residual saturation of CO₂. Images collected from the OSU ME Micro CT scanner were reconstructed to determine initial

and residual saturations at the center of the rock core. The same location was analyzed for each reconstructed image for consistency. Reconstructed images were generated using Octopus 8.5 and image processing was done with AVIZO.

CHAPTER IV: RESULTS AND DISCUSSIONS

Results obtained from the experiments are detailed in this section. Experiments performed aimed at developing a recipe to obtain specific viscosities, analyze surface tensions at different viscosities, and finally determine trapping capacity as a function of viscosity.

4.1 Obtaining desired viscosities

Volumetric mixing ratios of KI brine and the viscothickeners were used to obtain solutions of varying viscosities. Mixing ratios, which is the volume of viscothickener to volume of KI brine ranged from 0.05 to 0.33 for guar gum and 0.1 to 1 for glycerol.

Figure 6 shows dynamic viscosity of the solutions as a function of their mixing ratios. Results are reported to 90 % confidence. Higher viscosities are achieved with smaller mixing ratios of guar gum compared to mixing ratios of glycerol to achieve comparable viscosity changes.

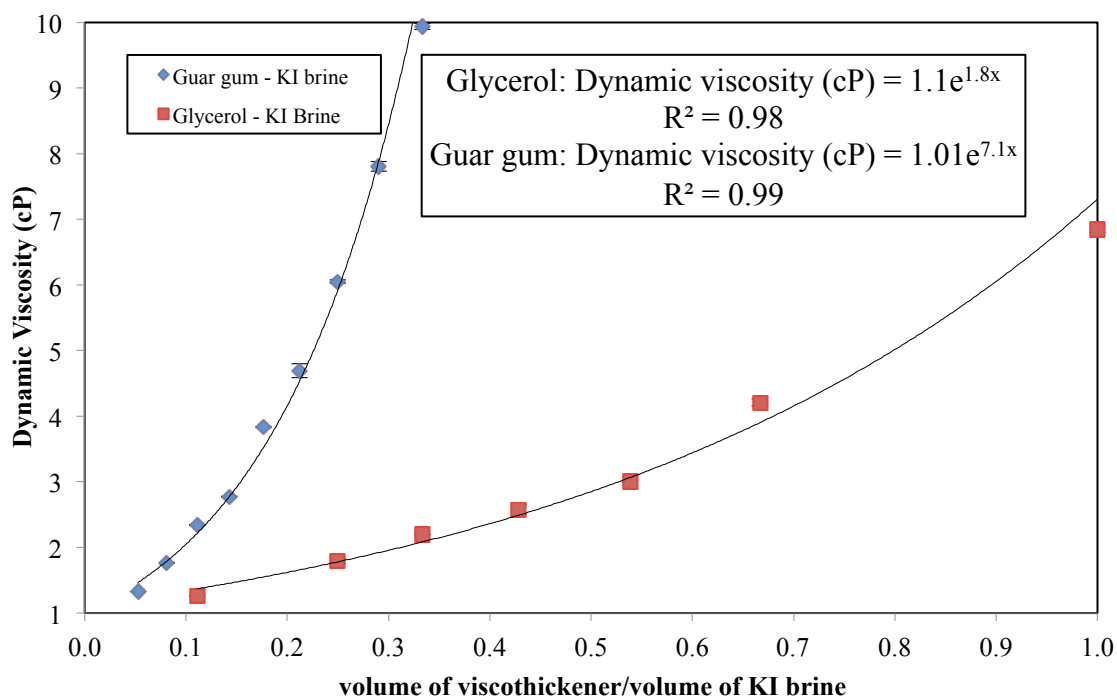


Figure 6: Dynamic viscosities of guar gum and glycerol when mixed with KI brine, as a function of the mixing ratios. Viscosity results are reported to 90 % confidence. Lower mixing ratios are needed for guar gum as compared to glycerol to achieve comparable viscosities.

Results from **Figure 6** show that obtaining desired viscosities with glycerol is easier than with guar gum. This is seen through the steepness of both slopes in the figure. The exponential functions are equations which can be used to determine appropriate mixing ratios to use in order to achieve desired viscosities.

4.2 Changes in surface tension

Surface tension measurements were taken at varying viscosities for both types of viscothickener solutions with KI brine. Apparent surface tensions were measured and a correction factor (F), see **Equation 2**, based on the manufacturer's specifications was used to obtain actual surface tensions. **Figure 7** shows the corrected surface tensions as a function of dynamic viscosity.

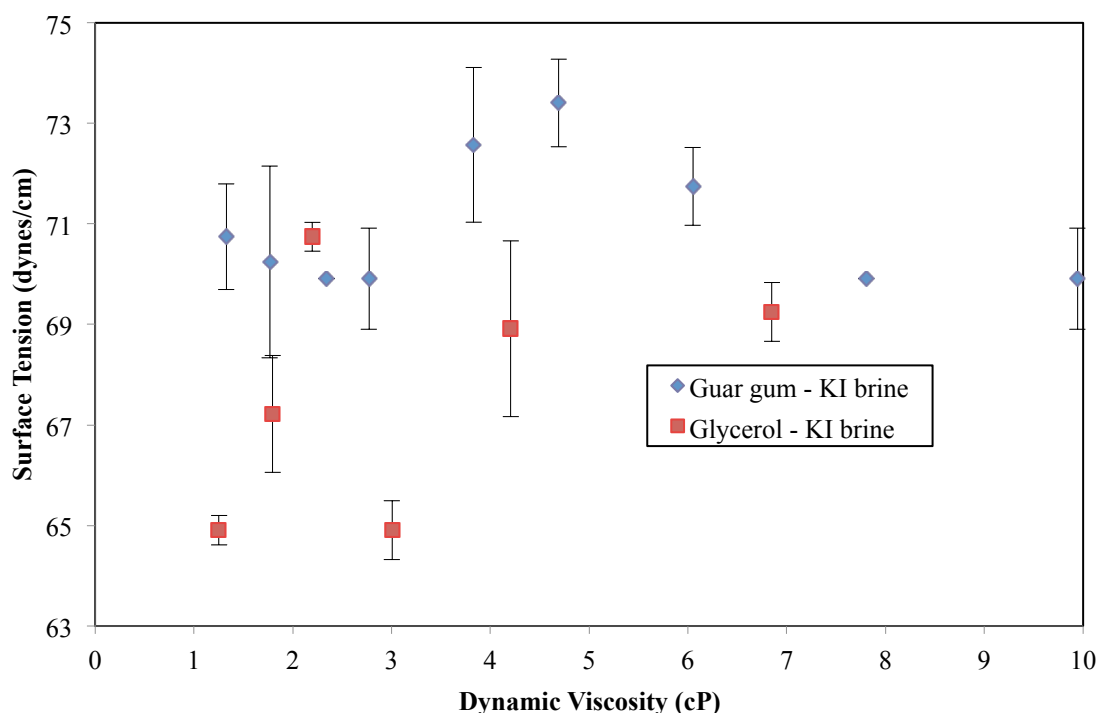


Figure 7: Surface tensions of KI brine as a function of dynamic viscosity, when viscosities were changed using guar gum and glycerol. Surface tension measurements are reported to 90 % confidence. Average surface tensions using guar gum are 71 ± 1.6 dynes/cm while average surface tensions with glycerol are 66 ± 1.8 dynes/cm.

Results presented in the **Figure 7** show that surface tensions obtained with guar gum were on average higher than those obtained with glycerol. Average surface tensions of KI brine with guar gum were 71 ± 1.6 dynes/cm while those obtained with glycerol were 66 ± 1.8 dynes/cm. Surface tensions with guar gum were closer to that of pure KI brine (72 dynes/cm) when compared to surface tensions of glycerol. When guar gum was mixed with KI brine and left to sit for a couple of days, disintegration of the solution was observed. Guar gum separated from KI brine to form small flocs in the solution. Surface tension measurements were taken over a series of days after a batch of guar gum and KI brine solution was prepared. Viscosities were randomly chosen and their surface tensions were measured. Surface tensions of solutions mixed with guar gum remain fairly constant as viscosity increased except between 3 and 8 cP. The change in surface tensions between those viscosities may be as a result of floc formation in guar

gum and KI brine solution. Separation of guar gum from KI brine may have changed surface composition and structure between air and liquid interface.

Apparent surface tension measurements with glycerol were also corrected using the correction factor (F). Results in **Figure 7** suggest that surface tensions obtained with glycerol increased with increasing viscosity and peaked at about 70 dynes/cm. The obtained results are within the surface tensions of pure glycerol (64 dynes/cm) and KI brine (72 dynes/cm).

4.3 Residual saturation

Guar gum and KI brine solutions at 2 cP, were injected into a rock core. Initial brine saturation and residual saturation of the non-wetting phase (CO₂) were determined from reconstruction of x-ray scans collected. Capillary numbers were calculated using **Equation 1**.

Figure 8 shows fractions of residual saturation measured as a function of the capillary number.

Circled points on the figure are the fractions of residual saturation obtained when using 2 cP guar gum and KI brine solution. The other data points were obtained without guar gum, when injection flowrates were varied. Higher fractions of residual saturation were observed when the viscosities were increased.

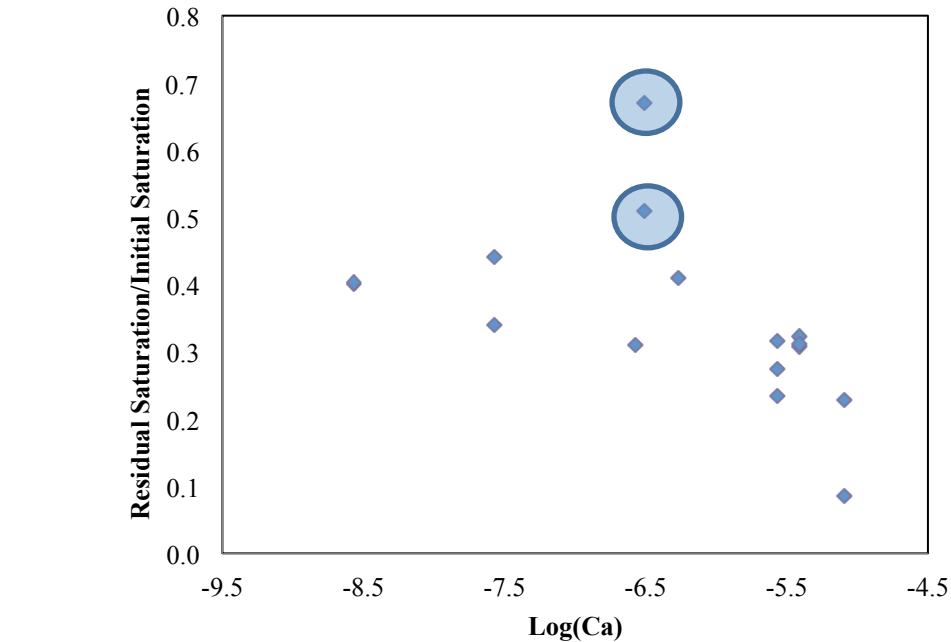


Figure 8: Fraction of residual saturation as a function of capillary number. Residual and initial saturations were obtained when solution was injected in rock cores, x-ray scans were taken and the images reconstructed. Circled points show effects with changes in viscosity using guar gum.

Higher than expected fractions of residual saturation may suggest that higher viscosities lead to increased trapping capacity. However, the formation of flocs in the guar gum and KI brine solution may be clogging up the rock pore and this may be the reason for higher fractions of residual saturation. In addition, the changes in surface tension may also be influencing the observed residual saturation. It is also important to note that the viscometer used (Cannon Ubbelohde Size 75) was designed to measure viscosities of Newtonian fluids [19]. A non-Newtonian fluid is formed using guar gum as the viscothickener. Higher viscosity solutions may have been created than was measured using the Cannon Ubbelohde Size 75, which may have also affected the residual saturation observed.

CHAPTER IV: RECOMMENDATIONS AND CONCLUSION

5.1 Recommendations

The equations shown in **Figure 6** can be used to obtain desired viscosities for guar gum and glycerol. Other viscometers such as a Turning Fork Vibration Viscometer [15] (specifically for non-Newtonian fluids) or a rheometer should be used to verify the obtained viscosities shown in **Figure 6**. Surface tension measurements for glycerol and guar gum should be repeated to verify variations obtained during the study. Fresh batches of guar gum and KI brine should be used for surface tension measurements in order to minimize the effect of floc formation.

Higher viscosity solutions of guar gum and glycerol should be injected into the rock core to verify whether higher viscosities increase residual trapping potential or whether the guar gum may be clogging the rocks.

Due to insufficient time, no experiments were done to change viscosity of non-wetting fluids. However the fluids suggested by Hatzikiriakos and Ochoa [14] should be used to change viscosities of the non-wetting fluid. The obtained solutions with varying viscosities should be injected into rock cores to determine their effect of residual trapping saturation.

5.2 Conclusion

In conclusion, this project developed methods for altering viscosity without changing interfacial tension, in support of a larger research project. The work established the required mixing ratios needed to obtain desired viscosities and the effects of viscosity on surface tension when guar gum and glycerol were used to increase viscosity. This project suggests that higher viscosities may increase residual trapping capacity and provides preliminary evidence of the role of viscous effects on capillary trapping, however more experiments are needed to draw conclusive results.

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