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Development of a Compact Core Flooding Apparatus for Matrix Acidizing Applications

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DEVELOPMENT OF A COMPACT CORE FLOODING APPARATUS FOR MATRIX ACIDIZING APPLICATIONS

A Thesis

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ABSTRACT

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Matrix Acidizing is defined as a well stimulation technique in which the acid is injected into the formation with the purpose of dissolving the minerals present and thus enhancing the permeability of the formation and facilitating the extraction of oil and/or gas from the formation. On carbonate formations, once the acid has been injected, wormholes and ramification paths form, and allow the fluid to flow primarily on the high permeability zones by following vugs, fractures and pores. HCl is commonly used for this process because of its quick reaction and fast rock dissolution. Matrix acidizing is one of the most utilized well stimulation techniques because of its low cost when compared to other techniques. It is known that there are four main parameters affecting the carbonate's dissolution performance: Injection rate of the acid, fluid type (viscosity), and acid concentration.

To conduct experiments simulating real field conditions, a core flooding laboratory setup was developed. Acid is injected at relatively high pressures using a continuous flow syringe pump. The acid and the brine are injected at pressures below the fracture pressure to avoid rupturing the carbonate.

Furthermore, to simulate the overburden pressure, self-weight of the soil or hydrostatic pressure, an overburden pressure pump was added. Finally, it is also important to add the back pressure exerted by the well, this with the use of a backpressure regulator.

The main goal of the laboratory development is to study, analyze and understand the optimum conditions for wormhole creation. System will be tested with multiple injection rates, back pressures, and overburden pressures to test for full functionality and reliability.

The core flooding apparatus was design to test at pressures as high as 5000 psi, stainless steel tubing, valves and fittings were utilized to support the corrosive nature of the Hydrochloric Acid. A compact design was developed as one of the main goals is to reduce the length of the tubing and thus reduce the amount of pressure drop.

DEDICATION

To my mother, my father, my wife, and everyone who helped me to be what I am and pursue my dreams.

ACKNOWLEDGMENTS

I would like to thank my committee chair, Dr. Pournik and my committee members, Dr. Lozano and Dr. Vasquez for their guidance and support.

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CHAPTER I

INTRODUCTION

Matrix acidizing has been a widely used acid diversion technique that allows oil extraction. It is one of the two stimulation techniques by multiple oil and gas companies. It consists of injecting a reactive acid into the well to dissolve the minerals present and increase its permeability by enlarging and connecting the pores of the reservoir. This process is achieved by injecting the fluid at pressures below rock fracture. Once injected, the acid/rock interaction triggers a series of reactions that lead to the formation of wormholes. These wormholes are defined as a narrow and long channel that connects and significantly enlarges the pores in the formation. Multiple parameters such as injection rate and volume are dependent on the permeability of the formation.

To enhance the matrix acidizing diversion technique, it is necessary to build a laboratory capable of simulating the real field and downhole conditions. This includes but is not limited to injection pressure, injection rate, injection volume, overburden pressure, back pressure, and temperature variations. Many core-flooding apparatus have been designed to simulate matrix acidizing technique in a laboratory, analyze, understand the process behavior, and thus develop new additives and diversion techniques for field applications. However, no research has been performed on the correct installation, setup, and usage of these laboratories. Although it might appear simple, a bad equipment configuration or test not performed correctly will lead to wrong

data and non-sense information when compared to the field. Multiple scenarios such as a bad drilled core sample, a bad core holder assembly, a missed valve configuration, and an overall bad testing quality have not been studied to explain the optimum testing conditions for a good quality laboratory testing. Furthermore, it is important to perform multiple functionality tests to ensure that the apparatus performance is optimum and is suitable for future research.

Oil and gas is still being considered as one of the dominant world industries, and will continue for multiple decades as most of the plastics, oil, fuel, and chemicals depend on the same raw material “petroleum”. As technology progress and the world thrives, the demand for raw materials is constantly increasing. Companies invest millions in research, and progress is constant on oil extraction enhancement. Additives such as surfactants, nanoparticle-based additives and gelled acids have been introduced in the las years as possible candidates to enhance matrix acidizing by allowing the fluid to flow through the less permeable paths and receive the adequate treatment. The design and development of a compact core flooding apparatus for matrix acidizing purposes will help for future research and development of multiple acid additives. It will serve as a base for understanding the principles of a core flooding set up and testing.

A detailed explanation of the components and equipment is provided along with the correct and incorrect testing parameters and scenarios. An expansion plan is provided as the lab is expected to get modifications and equipment additions as the technology changes. Multiple tested scenarios will describe the optimum testing conditions, the required maintenance and safety steps to enhance the laboratory’s outcome. Design also allows for testing in multiple rock types such as sandstone, limestone, dolomite and all the carbonate rocks. These experiments will serve as a basis to develop and study multiple fluids for wormhole enhancement as well as understanding heterogeneity effect and acidizing behavior on different scenarios.

CHAPTER II

LITERATURE REVIEW

2.1 Carbonate Matrix Acidizing

Matrix Acidizing is defined as a well stimulation technique in which the acid is injected into the formation with the purpose of dissolving the minerals present and thus enhancing the permeability of the formation and facilitating the extraction of oil and/or gas from the formation. Matrix acidizing has been utilized for more than 100 years as a process to remove the damage caused by drilling, process residues, completion and/or production operations clogging or blocking the pores near the wellbore. “Matrix acidizing refers to one of two stimulation processes in which acid is injected into the well penetrating the rock pores at pressures below fracture pressure” [1]. It is considered a cost-effective technique, however, sometimes delivers inconsistent outcomes due to the reservoir properties (porosity, permeability, heterogeneity).

Carbonate Matrix Acidizing consists in injecting acid (usually Hydrochloric Acid, HCl) into the wellbore between the pore pressure and the fracture pressure, otherwise, process will fail. Once the acid is being injected at optimum injection rate, it will travel across the formation from the wellbore to the reservoir without fracturing and/or damaging the formation. “The treatment is usually administered in a region approximately three to five feet around the wellbore” [2]. HCl is preferred over different acids because of its low cost and strong dissolution

reactions with calcite and dolomite. Usually, the hydrochloric acid concentration ranges from 5 to 28% m/m, having 15% m/m as the optimum HCl concentration. “Hydrochloric acid has advantages over other acids because it forms chloride salts, which are highly soluble in the aqueous phase and dissolve minerals like calcite, dolomite and siderite” [3].

Opposite to sandstones, in carbonates the affected region can extend up to several feet. The dissolution reactions of calcite ($CaCO_3$), dolomite ($CaMg([CO_3])_2$), and siderite ($FeCO_3$), with HCl acid may be expressed as follows:

$$2HCl + CaCO_3 \rightarrow CaCl_2 + CO_2 \uparrow + H_2O$$

$$4HCl + CaMg[CO_3]_2 \rightarrow CaCl_2 + MgCl_2 + 2CO_2 \uparrow + 2H_2O$$

$$2HCl + FeCO_3 \rightarrow FeCl_2 + CO_2 \uparrow + H_2O$$

On all three cases, the reactant products are soluble in water and do not cause any damage to the stimulation process. “Performance of acidizing of carbonates varies greatly depending on the application. The parameters that affect performance in carbonates are:

1. The concentration of calcite in the matrix
2. The acid injection rate
3. The reaction type (which is a function if acid type and temperature)
4. Heterogeneity” [4]

Through the years, multiple auxiliary chemicals have been developed to modify the acid solution injected to improve the acid dispersion in the formation. Since the creation of a

corrosion inhibitor as an additive, many other additives have been developed including surfactants, silicate agents, etc.

2.2 Worm hole propagation

Worm hole propagation refers to the continuous path that forms as the HCl is injected though the formation. “The whole process can be summarized as an acid-rock interaction with constant alteration to the porous structure of the rock through dissolution” [6]. There are different categories in which the dissolution patterns in carbonates can be classified: Wormhole, Uniform, or Compact. Wormhole propagation geometry heavily depends on the injection rate, porosity, and the kinetics of the surface reaction. Geometry of a wormhole is strongly affected by the matrix acidizing efficiency. Analysis shows that the path taken by the wormhole “depends on the acid transport ahead of the wormhole tip and the wormhole propagation preference in relatively large pores” [6]

“For regular acid, the patterns developed can be classified as one of the following:

- Compact or face dissolution in which most of the acid is spent near the rock face.
- Conical wormholes.
- Dominant wormholes.
- Ramified wormholes.
- Uniform dissolution” [5]

Testing performed by Wang, shows that at low injection rates, acid is spent at the core surface. If acid is injected at high rates, a multiple and highly ramified wormhole create.” At the optimum intermediate rate, a single small wormhole penetrates the core” [7].

2.3 Viscosity Controlled Acid (HCl)

Acid diversion technique has been on practice for more than 100 years, however, despite its longevity, there is still a huge room for improvement. In recent years, research has been performed towards acid modification, this will help to have a controlled acid diversion and achieve acid interaction in the low permeability zones. Addition of surfactants, nanoparticles and multiple additives create a benchmark between the conventional and an enhanced acidizing. For this research to continue its progress, it is crucial to have a laboratory to perform these experiments. Despite the development of a core flooding laboratory is mainly focused on performing research for an in-situ gelled acid, multiple different tests will be developed as the laboratory is suited for all testing types involving acid diversion.

2.3.1 In Situ Gelled Acids

As the HCl penetrates the carbonate formation, the flow diversion from a high-permeability region to a low-permeability region is key to a successful stimulation of carbonate reservoirs, especially for long horizontal and multilateral oil wells [8]. Usually, matrix acidizing involves the injection of regular HCl that flows through the high-permeability zones, leaving the low-permeability zones under-stimulated. In-situ gelled acids have been developed to obtain a more uniform stimulation. They consist of an acid soluble polymer, a pH buffer, cross-linkers, and breakers. In-situ gelled acids and viscoelastic surfactants function under the same principle. At very low pH, the acids present a relative low viscosity. As the pH increases to a determined value, the pH will increase by the action of the crosslinkers, leading to a gel formation with a significantly increased viscosity by one or two orders of magnitude. Once the pH limit has been reached, the polymer links are broken. “Several experimental studies have been performed to

understand the matrix acidization process in carbonate reservoirs with diverting acids, such as in-situ gelled acids and surfactant-based viscoelastic acids" [9].

In-situ gelled acid additives increase the viscosity of the fluid and retard the acid reaction with the formation, improving the treatment efficiency. A clear disadvantage of these gelled acids is associated with increasing the acid viscosity. Damage in the formation is developed due to the polymer entrapment or precipitation of cross-linker. "The behavior of the gelled acid is determined by temperature, which affects viscosity and acid reaction rate. Dilution of the acid with formation brine will reduce viscosity and diminish the effectiveness of the cross-linking reaction" [8]. Experiments performed with gelled acid shows that severe formation damage can result from the improper use of in-situ gelled acids. Once injected, the polymer will increase the viscosity of the modified acid which will reduce the diffusion rate of H+ from the bulk solution to the surface of the rock. "Polymer molecules can adsorb on the rock surface and form a barrier that reduces acid reaction with the rock. Finally, polymer solutions are non-Newtonian fluids with viscosity that decreases with shear rate (shear thinning behavior). Polymers can change the flow pattern close to surface of the rock, and therefore, affect the way the acid reacts with the rock". [10]

An experiment performed by Nasr El-Din [10] of a rotating disk instrument to measure the reaction rate of gelled acids with calcite rocks shows that the results obtained indicated that the apparent viscosity increased notably as the concentration of polymer increased from 0.5 to 1.5 wt%. On the other hand, there is significant decrease in the dissolution rates as the concentration of polymer was increased from 0.5 to 1.5 wt%, where there was no measure difference as the concentration increased to 2 wt%. Reverse and toroidal flows were noted within the rotational spends examined. The etching pattern on the surface of the disk depends, among

other factors, on the disk rotational speed and polymer concentration. The dissolution rate was found to be a function of temperature, rotational spend and polymer concentration.

A different experiment realized in University of Houston utilizing in-situ gelled acids showed that as a result of gel formation, the overall pressure drops increased and then decreased with the pore volume of the acid injected, on the other hand, on Newtonian fluids the pressure dropped constantly. On gelled acids, the optimum injection rate was proved to be smaller compared to Newtonian acids (HCl). Furthermore, gelling acids tend to create more branching with thinner wormholes as well as a better stimulation of the less-accessible low-permeability zones. [8]. One of the drawbacks of using gelled acid is that the gelling agents degrade in acid solution at temperature above 130 °F and they are not often used in matrix acidizing due to the injectivity loss by the high viscosity.

2.4 Nanoparticle based Viscosity Controlled Acid

Usually, carbonate formations are characterized to present a high degree of vertical heterogeneity with huge permeability and pore size variations. To obtain a uniform acid distribution, researchers developed an acid diversion system using nanoparticles as a retarded acid to manipulate injectivity into high permeability zone so the acid can flow into the low permeability zone. “Nanoparticles aggregate size distribution evolves with time, and once it spans pore space, gel structure is formed” [11]. Behavior of nanoparticles at different temperatures, pH, ionic strength, and salts has been studied experimentally in order to develop a system predicting the gelation kinetics of these nanoparticles. Despite the improvements on different surfactants and gelled acids, “the use of nanoparticle-based acids in acid diversion is proven to be more effective than the conventional diversion systems especially for harsh

reservoir conditions". The use of nanoparticle based gelled acids in matrix acidizing is intended to create continuous and uniform wormholes in all types of rock permeabilities.

There are two principal classes of silica nanoparticles that are widely used in dispersions: fumed silica and precipitated silica nanoparticles [11]. After silica nanoparticles are added to the gelled acid, the pseudo-solid shear-thinning gel can be controlled in pH along with the particle concentration. Interactions of nanoparticles and porous media include different mechanisms such as surface deposition, multi-particle plugging and single particle plugging that allow the silica to enhance fluid transportation in the formation.

On Abdelfatah's work, an acid diversion nanoparticle-based fluid was introduced by mixing HCl with nanoparticle fluid along with a crosslinker forming a gelatat between above point zero change of the nanoparticle. "The gel is formed at particle volume concentration as low as 1 vol%. The gelled acid performs like a power law fluid with a very small yield stress" [11]. He studied the effects of shear rate, temperature, pH and particle volume concentration of the diverted acid. On his testing, "Fumed metal oxide nanoparticles were used as pigments, viscosity adjusters, catalyst supports and fillers." He also used 7nm fumed Silica nanoparticles because of its high dispersion in aqueous phase. He described the process as "continuous flame hydrolysis of silicon tetrachloride". In which Silicon Tetrachloride was converted to gas phase and then reacted with intermediately formed water to produce silicon dioxide. On the multiple testing performed, three different crosslinkers, labeled as A, B, C were utilized. 1.25% vol% Silica was injected.

Table 1-Acid diversion in carbonates with nanoparticles based in situ Gelled acid [11]

pH	9% Crosslinker A	9% Crosslinker B	9% Crosslinker C
1	X	X	X
2	X	X	X
3	X	soft gel	X
4	X	Gel	X
5	X	Gel	X
6	X	Gel	X
7	X	Gel	X
8	X	Gel	X
9	X	Gel	X
10	soft gel	Gel	soft gel
11	soft gel	Gel	soft gel

The table above shows the results obtained from this experiment. It can be observed that crosslinker B formed a soft gel on pH 3 and gelled acid between pH 4 and pH 11. This gelation superiority was due to "higher charge density resulted in more deprotonates silanol groups on the surfaces at higher pH" [11].

A researcher Bang performed tests with silica nanoparticles dispersed in deionized water and blended with electrolytes to generate a stable gel correlated to solution pH and concentrations of MgCl2 electrolyte and silica particle. In his study "single and parallel core flood tests were implemented to study the transport behaviors of nanoparticle-based acids in porous media. The cores used in the experiments have a permeability range of 2 to 70 md." [12]. On his results, he states that the Mg2+ modified silica particles can create a gel almost instantly at volume fractions of Silicon Dioxide as low as 0.75 vol% at neutral pH due to aggregation into networks of silica particles. Furthermore, the temperature directly affects the rate of acid gelation. According to testing "Results of single core pattern flooding reveal that injection of the new nanoparticle-based formulations creates multiple wormholes and flow channels using both low (2md) and high (70md) permeability Indiana limestone core plugs". Some of the outcomes

from this experiment showed that the use of nanoparticle-based acid diversion into formations with low permeability will successfully divert the acid leading to the generation of continuous and multiple wormholes.

On his core flooding experiment, Bang utilized Indiana limestone cores with 1.5 in diameter by 6 in long and studied two different permeability ranges: 2-4 and 70-80 md with the purpose of studying the gel behavior and wormhole propagation at different permeabilities. “The objective of the core flooding experiments was to evaluate how nanoparticle propagate when fluid containing high volume fraction (0.75-1 vol%) with 15% HCl is injected” [12].

2.5 Core Flooding design for Matrix acidizing experimentation

Most of the existing core flooding apparatus tend to follow similar components as they are primarily designed for the same goals with a few variants in fluid composition, core sample sizes and parallel core flooding. Among its basic components the most important ones are the accumulators for both brine and acid, syringe pump, core holder, a back pressure regulator, an overburden pump to pressurize the sample in all directions, pressure transmitters for an accurate pressure difference reading and pressure gauges along the tubing to record the backpressure, the overburden pressure, and the inlet pressure. As temperature plays an important role in core flooding, a heating tape regulated with relays can also be added to simulate higher temperatures in the reservoirs.

Rock samples are usually obtained from drilling directly into the reservoir that is to be studied. A special core rotary drill is utilized to obtain the correct size for the cylinder’s diameter and length. The rock sample is then placed into the core holder and the system is then sealed. A refilling system usually consisting in a clear PVC container and compressed air injects the brine

and the prepared HCl into the different accumulators. Then the injection pump starts injecting hydraulic oil into the accumulators with the purpose of pushing the brine and the acid into the core holder. Pressure drop and flow rate are measured as the pump is injecting the fluid. The sample is previously compressed in all directions with the overburden pressure using hydraulic oil. Nitrogen is injected from the bottom of the core holder and with the aid of a backpressure regulator the pressure is maintained constant and thus simulating the reservoir conditions.

Core flooding is a widely used technique for testing multiple modified fluids and their interaction with rocks. The addition of additives, surfactants, and nanoparticles for testing in the laboratory have been helpful to progress, predict and enhance the field operations.

2.6 Basic core flooding requirements

2.6.1 Injection, overburden, and back pressure.

Pressure is the main component in matrix acidizing. Injection pressure should always be above backpressure and below overburden pressure. If back pressure is higher than the injection pressure, the produced carbon dioxide tends to stay in the sample and change from liquid to gas phase.

2.6.2 Temperature

Worldwide reservoirs present unique characteristics when regarding to altitude, pressure, and natural conditions. Temperature plays a significant role when matrix acidizing technique is applied. Rock permeability, porosity and field conditions are mainly impacted by temperature. It is well known that the permeability decreases as temperature increases. Yongming Li confirms

this statement after analyzing the effect of temperature field in acid diversion. He explained that despite multiple additives have been developed such as foams and VES, “their stability and strength are poor under high temperature and pressure” [13]. For the system to test at real downhole conditions and additive effectiveness, temperature should be able to increase. Heating tape can be added to the core holder to increase the overall system’s temperature.

2.6.3 Material requirements

The system is required to support pressures up to 5000 psi, relatively high temperatures and highly corrosive materials. It is known that stainless steel is considered a great corrosion resistant due to its chromium content, and the more chromium content the higher the resistance. Because of its higher concentration of both nickel and molybdenum, SS316 has a higher chloride’s pitting resistance than SS304. According to the stainless-steel laboratory corrosion data [13], and its study on resistance of stainless steel to chemical media, stainless steel is not considered as great material for HCl handling. It is expected to present a pitting corrosion of approximately 50 Mils per year (MPY). However, if a calculation is performed using this standard and with the 1/4” SS316 tubing with 0.065” wall thickness. According to Superior Fluid Solutions [15] a tube of these dimensions supports a pressure up to 10,200 psi. The lowest recommended wall thickness is 0.025” and supports a pressure up to 4000 psi while a 0.035” supports 5100 psi. Converting the 50 MPY into actual inches corrosion yields 0.05”. Splitting into months yield an approximate of 0.004166 inch per month of pipe corrosion. Using a 0.035” wall as the lowest permitted thickness for safety requirements, it would take an approximate of 7.2 months in continuous acid/tubing interaction to reach a 0.035” wall thickness and have a maximum pressure rating of 5100 psi. In other words, it would take 5,256 hrs. The experiments

performed are expected to last 30 minutes for acid injection and 30 minutes for tube flushing. Considering an average time of 1 hour per test and performing 3-5 tests a day it will require 1,051-1,752 days or a range from 2.9 to 4.8 years to require tubing maintenance and laboratory reparations. According to the American Iron and Steel Institute, 302 stainless steels with an acid concentration of 10.3% at room temperature will present a corrosion rate of 84 MPY [16]. If the same approach is utilized, it will take 617-1,028 days or 1.7 to 2.8 years of continuous testing before maintenance is required.

2.6.4 Core holder and accumulators

To divert acid in the rock at high pressures and temperature variations, a core holder is required for this task. There are multiple diameters and lengths, however, for this application, a 1” diameter by 6” length is utilized. A core holder is a cylindrical container designed to hold a core sample and apply multiple pressures [17]. Its multiple machined ports are connected to the tubing to allow fluid flow. For overburden pressure, a Viton sleeve surrounds the core sample and pressure is applied between the sleeve and the cylindrical stainless-steel enclosure. Injection pressure and back pressure are applied from top and bottom respectively and pressure can be monitored from the top, bottom, and sides.

2.6.5 Fluid and air leaks

System is required to support pressures as high as 5,000 psi and temperatures up to 250 ˚F. High pressure rated components and fittings must be purchased and properly installed to avoid any fluid or air leak. Air release procedures and valves will be designed to allow the system to release air if it gets trapped within the lines or components such as the accumulators.

This trapped air will cause erroneous data as air is compressible and pressure will significantly vary. Hydrochloric acid is an extremely dangerous chemical that will be used for testing; hence, components must be compatible with this acid to avoid any possible damage or system deterioration.

2.6.6 Laboratory mobility and design.

The core flooding setup was installed and adapted in a modified frame with the main purpose of being compact, and easy to transport as the frame should be easily pushed by one single person. Its design includes wheels and a 2 x 5 ft frame. There was also space and preparation for future modifications such as the addition of a second core holder, another syringe pump, or a fracking apparatus. Its compact design also enables the system to be easy to understand and allow everyone to use it. The apparatus was also designed to reduce the length of tubing utilized and the total curvature in the pipes to significantly reduce the amount of leftovers and dead volume. Pipes and fittings were fixed to the frame using U-clamps and heavy-duty bolts. Valves, pumps, and fittings were placed in locations that are easy to reach. As the lines were shortened as much as possible, the amount of wasted fluid was considerably reduced.

2.6.7 Safety and precautions

One of the main and most important goals was to design the laboratory to be operable in the safest way possible for any user without presenting any hazard even if the user is new to the setup. Two- and three-way valves were placed in locations that are easy for every user to reach. Multiple fail-safes were added, such as by-pass lines and a completely fixed setup. System was

built utilizing hex-cap screws and torqued to specific bolt specifications necessary for handling the fittings in case of a line failure.

2.7 Existing core flooding apparatus

Previous core flooding apparatus have been constructed in the past years to enhance matrix acidizing procedure. All previous laboratories follow a similar principle with small different operational modifications. The first laboratory was built by Javier N. Gomez [18] and his main focus was to "evaluate the rock heterogeneities, treatment conditions, and acidizing mechanisms". On his testing he showed that temperature plays an important role in acidizing as it activates the reaction rate of HF-HCl acid mixtures in sandstone. On his conclusion he stated that "The use of higher concentrations of HF at high temperatures cause consolidation of the matrix adversely affecting the final stimulation". He was able to test at different flow rates and proved that at higher flow rates, there is a better permeability response. Among his testing he found that at 100 ˚F, the optimum injection rate is 30 mL/min. He could also conclude that in carbonate rocks the injection rate directly affects the volume of acid required to breakthrough. Low flow rates would yield a more uniform pattern dissolution.

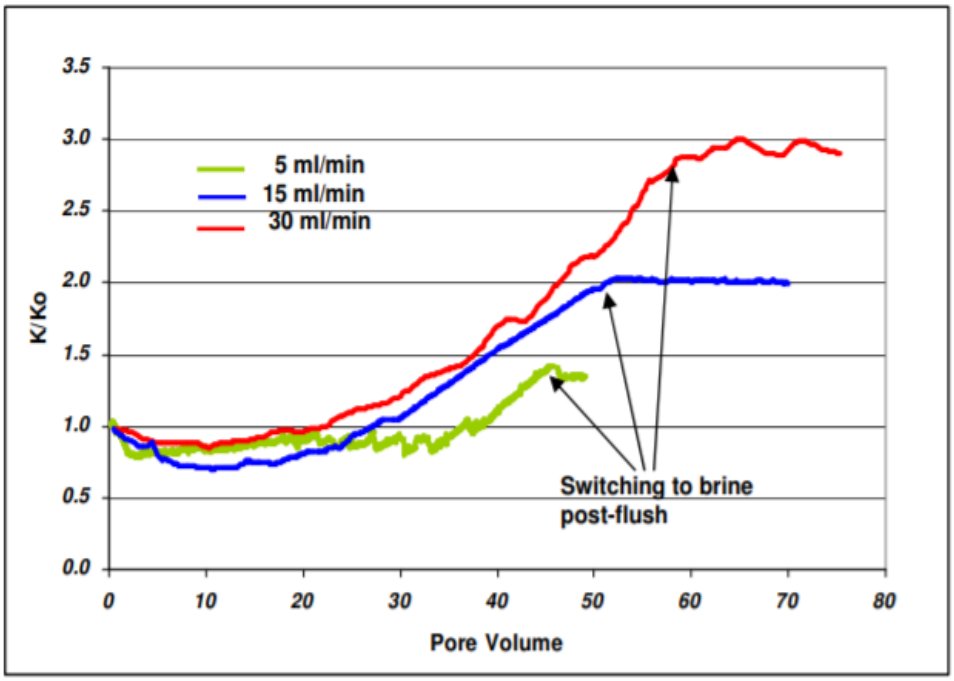

Figure 1. Effect of flow rate on acidizing sandstone at room temperature [18]

It can be visually observed in both figures how permeability response is improved once the flow rate is increase. This enhancement is produced because the residual products are flushed/displaced rapidly from the reaction place. On the other hand, low flow rate yields small permeability enhancement. Opposite to high flow rates, the slow displacement of the reacted minerals and formed precipitates create reservoir damage.

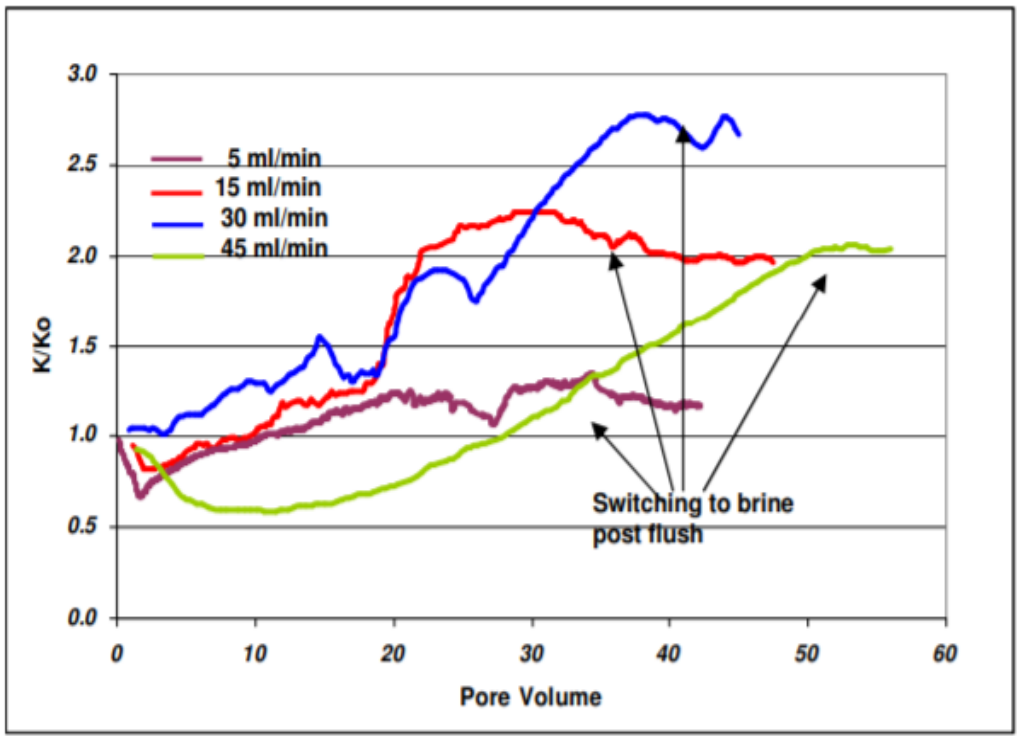

Figure 2. Effect of flow rate on acidizing Berea sandstone at 100 F

At higher temperature, results were similar with a few variations. From the chart, it is observed that despite higher flow rate (45 mL/min) was used, no further permeability response was achieved, leaving 30 mL/min as the optimal.

The apparatus was redesigned and expanded in Texas A&M University by Elizabeth Grabski [19] in 2012. Three different core holders were installed, a 1" x 6", a 1.5" x 20" and a 4" by 20". For injection pump a Teledyne 260D dual pump was also purchased. The total reported cost of equipment was $47,642 USD, however, many components such as the Syringe pump, which is the most expensive component are missing on her bill of materials. On her design, she followed a refilling system similar to what Nevito built; a refill tank that would inject the fluid to the accumulators with the aid of compressed air. She also was able to include temperature control and monitoring by adding heating tapes controlled by thermocouples connected to

LabVIEW software. An important aspect to consider is that on her design, the core holders had to be removed and placed back for every test. A full procedure had to be followed to load and unload the core sample including the hydraulic oil draining, the untightening of all the tubing ports and the core holder's frame disassembly. This procedure would take a huge amount of time and would lead to a huge room for error.

The laboratory was also expanded and improved by Vivek Gosh [20] in 2013, he was able to add a parallel core flooding setup and improve the acidizing procedure. After his implementations, the laboratory was rated for a pressure of 5,000 psi and 250 ˚F. He used a total of 36 valves to have the ability to run the parallel core floods at high pressures and temperatures. He also developed a well detailed user's manual for the laboratory. On his bill of materials, he reported a total equipment cost of $121,130.49 USD. Core holders had to be removed and placed back for every test as well. As they tend to be heavy and fragile, the best method was to design a system for core load/unload without core holder movement. On his experiments he was able to calculate and develop all the equations to obtain the required data for core sample's analysis. He could successfully provide examples of the calculations and procedures to obtain pore volume, permeability, porosity, pore volume to break through and interstitial volume. He also developed a core flooding data sheet to have summarize all the information and have a better data analysis.

Core Flooding Data Sheet

Core#	1	Date	April 18, 2013
Core diameter	1 inch		
Core length	3.75 inch		
Porosity Measurement		Acid Core Flooding	
Weight (Dry)	111 g	Temperature	140 °F
Weight (Sat)	118 g		
Porosity	14.5%	Acid Injection Rate	2 mL/min
Pore Volume	cm^3	Interstitial Velocity	2.72 cm/min
Permeability Measurement			
Brine Injection Rate	10 mL/min	Volume of Pipe to Core	27.90 cm^3
Pressure Differential	1608 psi		
Permeability	2.86 md	Time for Acid to Get the Core	836.9 seconds
Acid Formulation	15% wt HCl	Time for Acid Injection	2004.0 seconds
Water	626.94 mL		
HCl	373.06 mL	Pore Volume Break Through	5.56 Pore volume

Figure 3. Core flooding data sheet [20]

2.8 Objective

Core flooding is not a common apparatus available in research labs. However, it is extremely helpful as the Oil and Gas industry keeps growing and innovating. Despite renewable energies have been arising in the last years, oil and gas industry is expected to still be primordial for the world's progress within the next decades. Research on additives such as viscoelastic surfactants and nanoparticle addition to the acid have helped to facilitate and enhance the oil extraction, however there is still a lot of potential progress within the field. The use of in-situ gelled acids with the use of nanoparticles is a research topic that has potential progress within this field. Although years of research have been performed with the use of a core flooding setup, there is no research and testing available towards the explanation of the laboratory usage. Hence, there is no proof that most of the data obtained from testing is 100% accurate. The main purpose of this setup is to test multiple rock and laboratory conditions such as different sample sizes, shapes, and forms to compare and analyze results.

CHAPTER III

LAB EQUIPMENT AND PARTS

3.1 Equipment utilized

Before constructing the lab, it was necessary to perform a literature review in regards of the requirements for core flooding matrix acidizing test. Different existing core flooding setups as well as different equipment for accomplishing the required specifications were purchased. Quotes from different companies and different brands were requested. The equipment was selected based on the cost, quality and required specifications. The system was designed to satisfy a 5,000 psi of overall pressure. The maximum allowed temperature at maximum pressure is 250 ˚F. Stainless steel fittings, valves and tubing was utilized as it is strong against HCl corrosion. To have a total fluid flow control a 19 valve (two way and three way) system was designed. As only one core holder was purchased and assembled, the preparation for a lab expansion was planned and it will only require small modifications if necessary. This chapter will describe and explain the use of all the laboratory components as well as their role in the core flooding testing. Also, fluid flow will be explained and valve position for a successful test. Finally, the instructions and preparation for a laboratory expansion for further testing will be explained. The utilized components are as follows:

1. Erlenmeyer flasks
2. Syringe pump

3. Relief valve
4. 1/8 outer diameter stainless steel tubing
5. ¼ outer diameter stainless steel tubing
6. Hydraulic Oil
7. Two-way valve
8. Three-way valve
9. Stainless steel T-union
10. Accumulators
11. Metering Pump
12. Pressure gauges
13. Stainless Steel union cross
14. Core holder
15. Pressure transducers
16. Back pressure regulator
17. Nitrogen tank
18. Overburden pressure pump
19. Heating tape
20. Fittings
21. Data acquisition system
22. Frame

3.2 Core Flooding Schematics

Before proceeding to assembly, a detailed schematics with all the required components and equipment is described. The accommodation of the items is dictated by the fluid sequence requirements as well as the frame design. A short overview, system requirements involved to connect the injection and metering pump to the top and bottom of the accumulators to pump fluid in and out. From the accumulators, fluid travels directly to the core holder for rock stimulation. Pressure transducers are then connected to the inlet, center, and outlet of the core holder to obtain the real time pressure. Overburden pump is also connected to the core holder to provide pressure to the core sample. Finally, back pressure regulator is placed below the core holder to simulate the push-back pressure encountered downhole.

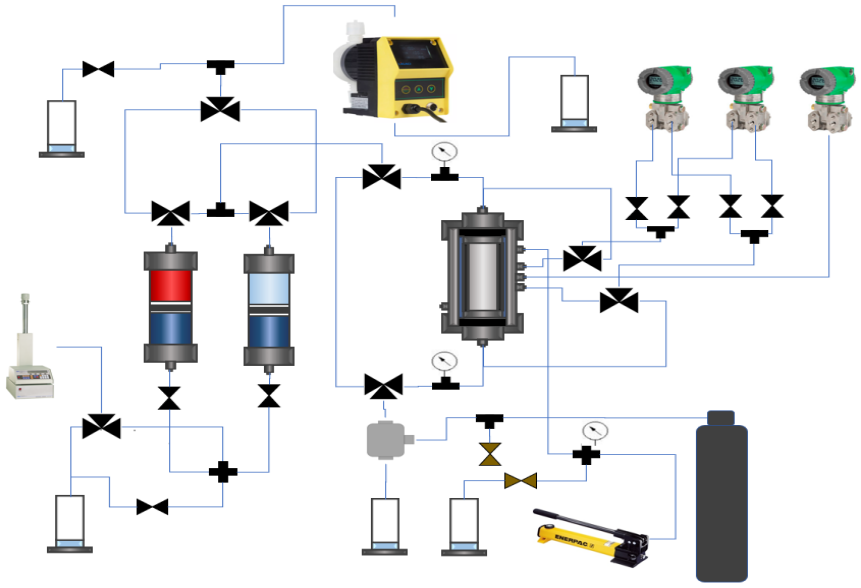

Figure 4. Core Flooding apparatus schematics

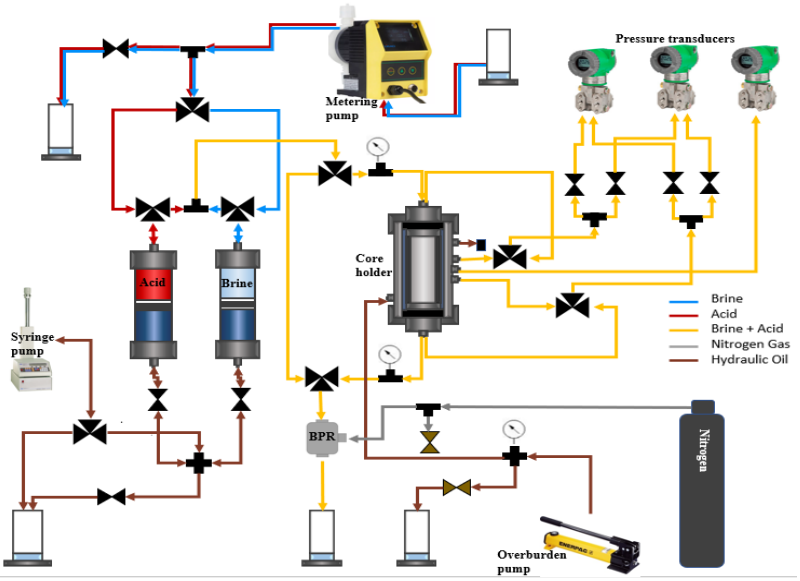

Figure 5. Core flooding schematics for flow direction.

Figure 5 shows the fluid directions through the tubing. The utilization of two- and three-way valves allow an easy fluid manipulation. Color code and arrow directions describe the different fluids that pass through the different tubing sections. Valve manipulations for the different laboratory operations are described in chapter 4.

3.3 Bill of Materials

A complete bill of materials is developed to manage and take decisions accomplishing both the system requirements and the available budget. Quotes from different vendors were acquired and after a careful analysis and a schematics development materials described in table 2 were purchased and installed.

Table 2. Bill of materials

Item	Material	Pressure rating	Temperature Celsius	Fitting Type	Fitting size	Part No./Model	Quantity	Price
EQUIPMENT AND COMPONENTS								
Syringe Pump		7500		FNPT	1/8"	260D	1	$13,523.50
Accumulator	SS 316	5000	250	FNPT	1/4"		2	$2,730.00
Metering Pump	PVC	150		MNPT	1/4"	JLM-S0110-PVC	1	$345.00
Hassler type core holder	SS 316	5000	300	Tubing	1/8"		1	$7,110.00
Pressure Gauges	SS 316	5000	150	MNPT	1/4"	4FML7	3	$22.41
Differencial pressure transducer 0-200 psi		200		FNPT	1/4"	IDP10S	1	$1,763.25
Differencial pressure transducer 0-2000 psi		2000		FNPT	1/4"	IDP10S	1	$1,838.03
Absolute pressure transducer 0-5000		5000		FNPT	1/4"	IGP10S	1	$1,697.60
Back pressure regulator	SS 316	6000	150	FNPT	1/4"	U6L2SNN8-NSGP6000T150K40LLKB	1	$1,946.97
Nitrogen tank		3000		FNPT	1/2"		1	$10.00
Nitrogen tank regulator				MNPT	1/2"		1	$479.00
Overburden pressure pump		10000		FNPT	3/8"	P392	1	$504.02
Erlenmeyer flask							5	$45.00
Power source							1	$90.80
Data acquisition						USB 6002	1	$447.00
Diamond core Drill							1	$1,620.00
VALVES, TUBING AND FITTINGS								
1/8" stainless steel tubing	SS 316	5000	220	Tubing	1/8"		4 ft	$0.00
1/4" stainless steel tubing	SS 316	5000	220	Tubing	1/4"		40 ft	$216.80
Two-way ball valve	Cast iron	7500		FNPT	1/4"		7	$99.75
Two-way ball valve	SS 316	6000	350	FNPT	1/4"	SBVF3602-F-4N-S316	1	$65.00
Three-way ball valve	Cast iron	7500		FNPT	1/4"		3	$51.90
Three-way ball valve	SS 316	6000	350	FNPT	1/4"	SBVF36023-F-4N-S316	5	$325.00
Needle valve	SS 316			FNPT	1/4"		2	$230.00
Relief valve	SS 316	6000		FNPT	1/4"	SRVH-S-4-S3	1	$127.50
Union cross (compression fitting)	SS 316		649	Tubing	1/4"	SUC-4-S316	2	$60.86
Union tee (compression fitting)	SS 316		649	Tubing	1/4"	SUT-4-S316	7	$117.81
Male adaptor (NPT to compression fitting)	SS 316		649	1/4" Tubing to 1/4" MNPT		SMC4-4N-S3	55	$289.85
Female adaptor (NPT to compression fitting)	SS 316		649	1/4" Tubing to		SFC4-4N-S3	5	
Male adaptor (NPT to compression fitting)	SS 316		649	1/4" Tubing to		SMC4-6N-S3	1	
Check valve (compression fitting)	SS 316	6000	649	Tubing	1/4"	SHCV1-S-4-2	2	$88.06
Reducing union (compression fitting)	SS 316		649	1/4" Tubing to 1/8" Tubing		SRU4-2-S31	4	
Male reducing adaptor (NPT to compression fitting)	SS 316		649	Tubing to 1/8"		SMC4-2N-S3	7	
TOTAL								$35,845.11

3.4 Core Holder

The core holder is considered one of the main components of the assembly, it consists of a special cylinder with multiple inlets and outlets. The drilled core sample (with dimensions of 1” diameter and 6” length) is placed inside and the core holder allows for multiple pressures to go through. Once the sample is placed inside, a Viton sleeve surrounds the entire sample and overburden pressure is applied by the hydraulic oil. Injection pressure is applied from the top with the syringe pump, and the back pressure is applied from the bottom with nitrogen gas and a back pressure regulator. The core holder consists of nine different inlets/outlets. From the two inlets on the top, one is utilized for the fluid inlet and the second for the high-pressure inlet of the differential pressure transducers.

The two outlets on the bottom are utilized for the fluid outlet and for the low-pressure inlet of the pressure transducers. Both top and bottom ports have a 1/8” tubing that connects to a 1/4” to 1/8” coupling reduction. The three ports in the middle are utilized as outlets for pressure measurements. The one on top connects to the high-pressure inlet of the differential pressure transmitter. The outlet in the middle connects to the inlet of the absolute pressure transmitter and finally, the bottom outlet connects to the low-pressure inlet of the pressure transmitters. The three ports are connected with a 1/4” MNTP to 1/4” OD tubing outlet. The last two ports on the side are utilized for the overburden pressure, the one on top is utilized as the inlet for the hydraulic oil while the bottom one connects to a relief valve. Overburden pressure ports are connected to the core holder with a 1/8” MNTP to 1/4” OD tubing connector.

The Hassler type core holder was designed to support pressures up to 5000 psi and temperatures as high as 300 ˚F. It was purchased from Phoenix Instruments manufacturer, and it is made of Hastelloy C276.

Figure 6. Core Holder assembly and components

3.5 Syringe pump

To simulate high and steady injection pressures, a Teledyne ISCO syringe pump is utilized as it provides high precision injection rate and injection pressure. It is a 260D series pump, designed to handle supercritical fluids and refilling under high pressure. It has a maximum injection pressure of 7,500 psi (517 bar) and a maximum injection rate of 107 mL/min.

The specifications provided by Teledyne are as follows:

Table 3. Teledyne ISCO 260D specifications [21]

Capacity	266 mL
Flow Range	0.001 to 107 mL/min
Flow Accuracy	0.5% of setpoint
Pressure Range	10 to 7,500 psi (0.7 to 517 bar)
Standard Pressure Accuracy	0.5% Full Scale
Wetted Materials	Nitronic 50, PTFE, Hastelloy C-276
Plumbing Ports	1/8" Valco
Operating Temperature	5 to 40 °C Ambient
Power Requirements	100 VAC, 117 VAC, 234 VAC, 50/60 Hz

It consists of two components, the pump and a digital controller that allows the user to easily navigate through the different options such as constant flow, constant pressure, maximum and minimum flow rate, etc. The minimum flow rate allowed is 0.001 mL/min. It is recommended to set the maximum injection pressure up to 4,000 psi as the entire system is rated for 5,000 psi. This to avoid any possible damage or system failure. As the pump can absorb/inject fluid from both ports, only one was utilized and the other was sealed. With the use of a three-way ball valve, the port located at the top of the pump serves as inlet/outlet for fluid refill and injection. The pump's port is a 1/8" MNPT to 1/8" tube connector. Furthermore, a 1/4" to 1/8" tubing compression union was utilized to connect it into the system.

If further expansion is desired, the pump is designed to support a dual pump configuration or up to four pumps configured in series. Despite the pump is designed to handle supercritical fluids due to its Hastelloy components Teledyne recommends the use of hydraulic oil or distilled water to avoid future corrosion or rust caused by the water or corrosive agents. It is recommended to change the hydraulic oil after 10 experiments as it will eventually get impurities and acid/brine leftovers from the accumulators.

Figure 7. Teledyne ISCO 260D syringe pump

3.6 Overburden pump

Overburden pump is essential to apply pressure in the core sample's body. An ENERPAC P392 two speed hand pump was purchased. It has a capacity of 0.9L with a maximum operating pressure of 10,000 psi [22]. It has a lightweight and a compact design that allows it to fit into the compact laboratory design. It has a 3/8"-18 FNPT port. To connect to the system, a 3/8" MNPT to 1/4" tubing connector was utilized. The hand pump provides overburden pressure to the core sample as it pumps the oil between the core holder and the Viton sleeve. It is important to denote that for a successful testing, and simulating real world conditions, overburden pressure should always be kept at least 300 psi above the injection pressure. Failure to maintain this requirement will result in the fluid passing through the sides, between the Viton sleeve and the sample and not through the core sample. The pump is connected to the core holder, a bleed valve and to a pressure gauge. It is important to constantly monitor the overburden pressure as it tends to drop with time or increase with a temperature rise. Finally, to release the pump's applied pressure, opening the bleed valve will allow the hydraulic oil to flow into a beaker. It is recommended to check the pump's oil level before every experiment to ensure that is full. To refill it, open the cap that placed on the rear top of the pump and with the aid of a funnel fill with ENERPAC HF hydraulic oil.

Figure 8. ENERPAC overburden pump

3.7 Accumulators

Two accumulators were utilized as fluid storing elements. They consist of a cylinder with a Teflon piston that moves up and down depending on the direction of the pressure being applied. Its main purpose is to store the brine and the hydrochloric acid before being injected into the core holder. They have a capacity of 275 mL each. They have a 1/4” FNPT port on top and bottom. To connect to the tubing, a 1/4” MNPT to 1/4” tubing connector was utilized. They are made of stainless steel and rated for 5,000 psi. Both accumulators were placed vertically and on the bottom hydraulic oil is injected with the syringe pump while acid and brine are supplied from the top with a metering pump. The pressure required to move the piston is 110 psi.

It is important to test the accumulators for air present in the system. Despite it was designed to not allow air to get in, if a wrong valve configuration is utilized, air can fill the accumulators and will cause erroneous data and results. As air is compressible, it will cause pressure fluctuations and bad readings by the pressure gauges. Furthermore, the injection rate and pressure will not be consistent, and the ratio of inlet/outlet fluid might vary. If any inconsistency is observed an air release procedure should be performed.


Figure 9. Acid and brine accumulators

3.8 Metering Pump and refilling system.

A LIGAO solenoid metering pump was purchased to refill both accumulators with brine and acid respectively. It is a JLM-S signal dosing pump with a microprocessor-based control board. The LED screen displays the current flow rate and the accumulated flow pumped. Its capacity extends up to 1.0 LPH with a maximum injection pressure of 145 psi. Amongst its main components are PTFE for the diaphragm and the valve seat, Viton for the O-ring, and PVC for the valve body. PTFE is known for its excellence in handling the most aggressive acids and corrosive materials.

The refilling system consists of the metering pump connected to a three-way ball valve that determines if the fluid goes into the first or the second accumulator. A second three-way ball valve allows the fluid to get into the accumulator for the refilling. A check valve is located between both valves to prevent fluid going towards the metering pump in case the valve configuration is wrong while injection pressure is applied. It is strongly recommended to pump

the acid first. Once the acid is refilled, open the two-way ball valve on the refilling system and pump water for at least one minute, this will clean the tubing and prepare the system for the brine refilling.

Figure 10. Metering Pump

3.9 Back Pressure Regulator

To fulfill reservoir conditions, a back pressure regulator was installed at the outlet of the core holder to hold pressure as the fluid pass through the core sample. In down hole conditions, back pressure keeps the carbon dioxide in liquid form as the acid-core reaction happens. Absence of this pressure lead to carbon dioxide gas formation and differential pressure increase. This two-phase effect is undesirable as it will lead to erroneous testing and the fines migration phenomenon. Back pressure is required to be 300 – 400 psi below the overburden pressure and the injection pressure. An EQUILIBAR BPR model U6L was purchased and utilized for the assembly. It has a range up to 6,000 psi and can perform at temperatures of to 150 ˚C. It was designed to hold a stable pressure across the flow range. It can also work with multiple phases

such as gas, liquid and/or multiphase fluids. It is capable of withstanding highly aggressive chemicals. Wetted parts are built from Stainless Steel 316L while the O-Rings are from Viton (FKM) and the diaphragm is from PTFE/Glass Laminate. It has an inlet/outlet port size of 1/4" FNPT while the reference port size (the one connected to the back pressure source) has a 1/8" FNTP. It's flow coefficient (Cv) ranges from 1E-09 to 0.05 [23].

Figure 11. Back pressure regulator

Figure 11 shows the exploded view of the model's construction, the diaphragm is sealed by two O-rings and placed between main body and the reference cap. The BPR is set to hold the process at a 1:1 ratio, this means that only when the set (injection) pressure exceeds the dome (nitrogen) pressure, the diaphragm will open, and flow will be released through the BPR.

3.10 Pressure gauges

To constantly monitor the pressure across the entire system, pressure gauges are located near the core's holder inlet and outlet to compare the pressure with the displayed by the pressure transducers. Also, a pressure gauge for the overburden pressure is required to keep the pressure at the required level. Installed pressure gauges have a rage from 0-5000 psi as the system is not intended to surpass that pressure. The main component for the wetted parts is stainless steel 316

and plastic for the front cover. It comes with a 1/4” MNPT connection. A 1/4” FNPT to 1/4” compression fitting is required to connect it to the system.

Figure 12. Pressure gauge

3.11 Valves

To accomplish fluid flow and pressure control, a set of two and three way-ball valves along with Needle and relief valves were implemented. A total of 19 valves were utilized including 1 relief valve, 2 needle valves, 8 two way-ball valves and 8 three way-ball valves. Two different types of valves were utilized as not all the valves are exposed to Hydrochloric acid.

3.11.1 Two way-ball valve.

Two different models were purchased, as many valves only interact with hydraulic oil. The only requirement is to support the injection pressure. Seven hydraulic two-way ball valves from TOOLOTS were installed. They are rated for 7520 psi working pressure and a temperature range from 14 to 185 ˚F. The main body is cast Iron Steel with two 1/4” FNPT connections. The second model is a SBVF360 series valve from superior fluids. As it is required to directly interact with acid, its main body is composed of Stainless Steel 316 with a pressure rating of 6000 psi and a working temperature range of -65 to 350 ˚F. A 1/4” FNPT connection was also installed. These valves are open-closed configuration. For both models, the valve is in the open

position when the handle is parallel to the body. In other words, the handle is in the same direction as the body. To change the configuration to close, it is necessary to rotate the handle 90 degrees, so the handle is now perpendicular to the valve's body.

Figure 13. Two-way ball valve

3.11.2 Three way-ball valve

Two different models were purchased for three way-ball valves. Three valves from TOOLOTS were utilized with a pressure rating of 7250 psi and a temperature working range from 14 to 185 ˚F. It comes with three 1/4" NPT connections and a two L-shape configuration for fluid flow. For this model, the handle has two different positions.

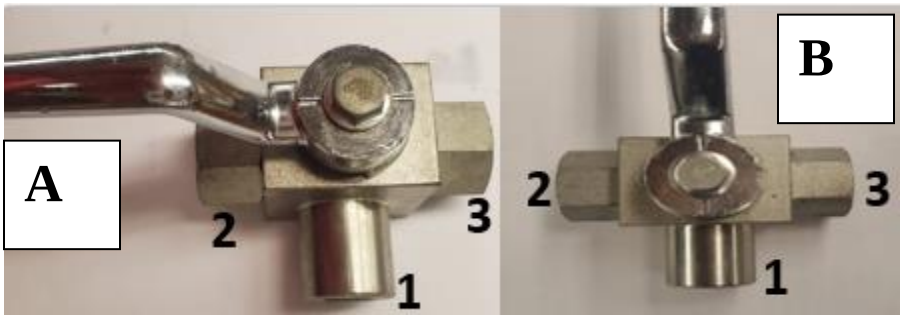

Figure 14. Cast Iron three-way ball valve

Figure 14 shows the three ports and the two handle configurations, when the handle is in position A, parallel to the body, the fluid will flow in either direction involving ports 1 and 3. When the handle is rotated 90 degrees to position B, in a perpendicular direction with respect to the body, fluid will flow in either direction involving ports 1 and 2.

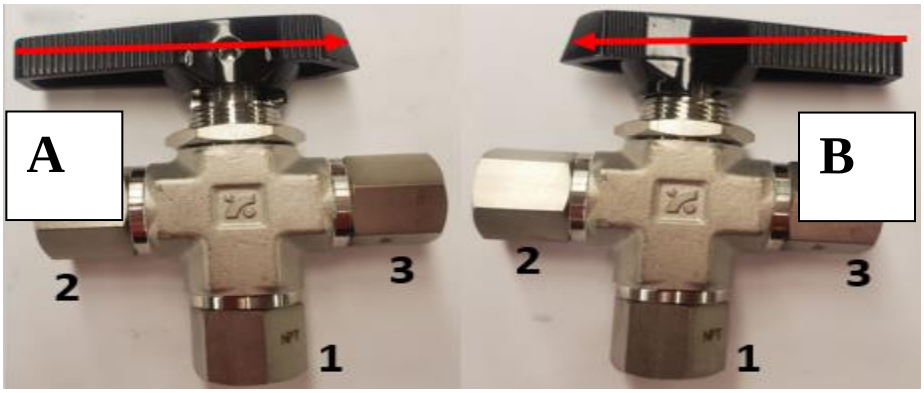

Figure 15. Stainless Steel three-way ball valve

The second model involves five three-way SBVF360 ball valve series. They can withstand up to 6000 psi and a temperature range from -65 to 350 ˚F. As they are required to interact with the acid, they are built from forged stainless steel 316 with all three ports having a 1/4” FNPT connection. Figure 15 shows the two handle configurations, in this model, the handle presents an arrow shape with a long and a short tip. If fluid is intended to flow in through ports 1 and 2, the arrow should be pointing towards port 2. In other words, the small tip should be above the port 2. If rotated 90 degrees, the valve will be in the closed position and thus not allowing the fluid to pass through any of the ports. If rotated 180 degrees, the valve will allow fluid to pass through ports 1 and 3. It will be observed that the tip is now above port 3 and the arrow will be pointing towards it.

3.11.3 High Pressure relief valve and spring replacement

Overburden pressure plays a primary role in core flooding. As temperature is intended to increase/decrease, overburden pressure also increases. Also, as the hand pump is user operated manually, non-desired extra pressure can be applied by the user. To override this constraint, a relief valve is configured to release the extra oil volume. A SRVH series high pressure relief valve from superior fluid solutions was installed. It has a maximum working pressure of 6000 psi and a cracking pressure range from 50 to 6000 psi. It is built primarily from SS 316 and has two 1/4" tubing compression connections. There are seven different pressure ranges controlled by different springs.

Table 4. Relief valve spring configuration

Spring code	Spring color	Set pressure range (psig)	Turns from initial position
A	Blue	50 to 350	9
B	Yellow	350 to 750	8.5
C	Purple	750 to 1500	9
D	Orange	1500 to 2250	6
E	Brown	2250 to 3000	6
F	White	3000 to 4000	6
G	Red	4000 to 5000	6
H	Green	5000 to 6000	6

As different parameters are utilized for matrix acidizing, pressures also vary. Table 4 shows the different springs that can be utilized to achieve the desired pressure range. For a spring replacement follow the next steps:

1. Remove the lock wire from the cap and the body (Figure step 1)
2. With a wrench, slowly loosen both the lock nut and the cap (Figure step 2)
3. Remove the cap along with the spring and the small cylindrical support (Figure step 3)
4. Insert the new spring along with the support.
5. Replace the label with the respective spring's label
6. Tighten the cap to the initial position and start counting the number of turns to the desired pressure.
7. Test the pressure and adjust to desired. Tighten to increase pressure and loosen cap for decrease in pressure.
8. Example, desired pressure is 2,000 psi. Install orange spring and rotate 4-360 turns. As the pressure range is from 1500 to 2250 psi and there is a total of 6 turns, every turn is 125 psi approximately.

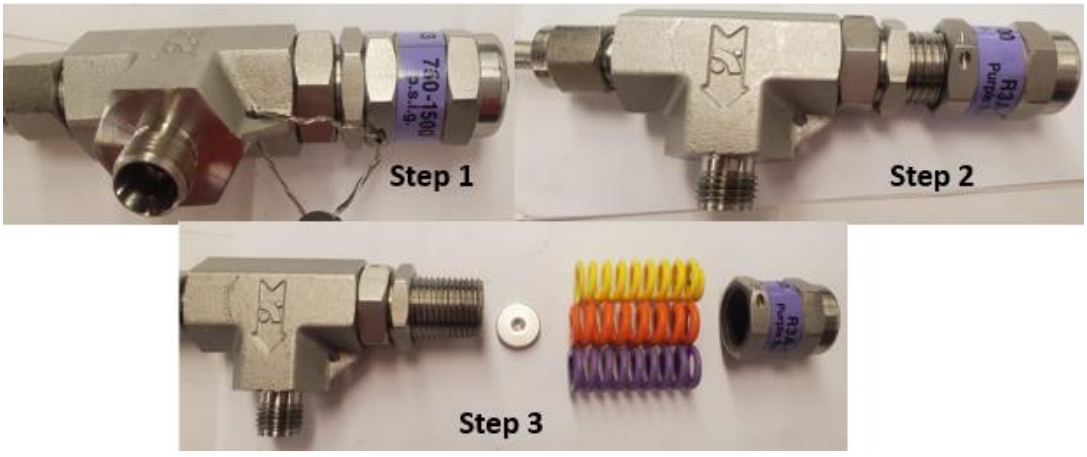

Figure 16. Relief valve spring replacement

3.12 Fittings, unions, and tubing

The laboratory consists of a total of 34 ft of 1/4” OD stainless steel tubing with a wall thickness of 0.065” and a pressure rating of 10,200 psi. All the components are assembled with SS tubing. To achieve a compact design, and the best component placement setup, most of the tubing was bended and shaped into the desired curvatures to connect the equipment. Curvatures were achieved with a pipe bender and wood molds.

As the laboratory consists of equipment of different sizes and connections, fittings and unions connect the components to the tubing and to the next component. The laboratory involves two different connection types, compression fittings and NPT (National Pipe Thread) fittings. Compression fittings connect the tubing straight into the fitting, to achieve this, multiple components are required, a nut, a ring, and a ferrule. As the nut is tightened, the ring and the ferrule compress the pipe and constraints its movement. Ferrules and rings can only be used once. Despite the nut is removed from the tubing, both components will still be fastened to the tubing. Valves, accumulators, pumps, and pressure transducers are connected through National Pipe Tread configuration. Most of the components have whether male or female National Pipe Tread configuration. As NTP does not work as the compression unions, to securely attach and prevent any leak, Teflon thread seal tape seals and prevents any fluid leakage.

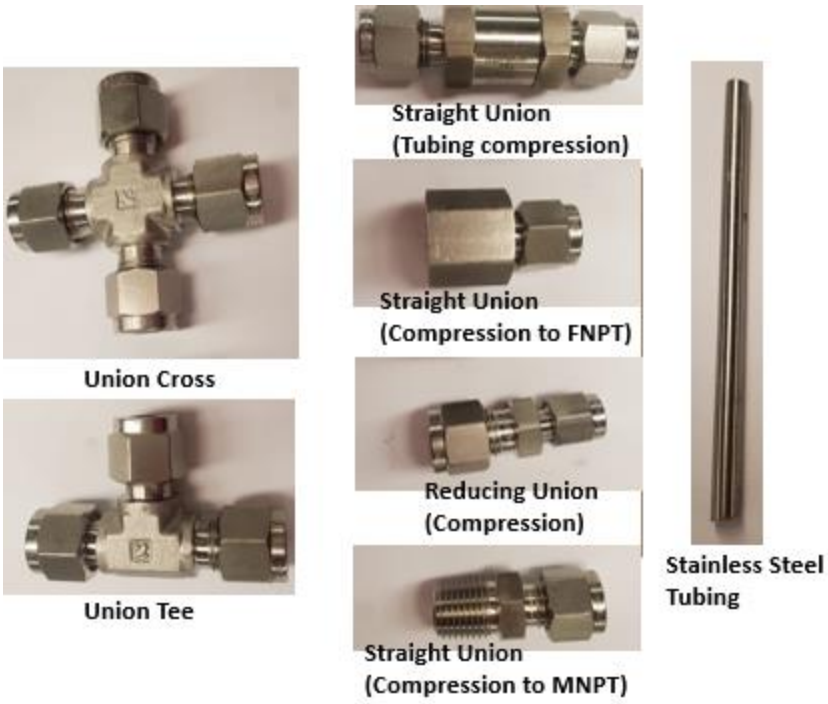

Figure 17. Unions, fittings, and tubing

If a new fitting is added to the system, to correctly perform the connections for a compression connection, the procedure is as follows:

1. Locate a clamp and secure the fitting in a position with free nut movement.
2. Confirm that the fitting has the nut, ring, and ferrule in the right orientation.
3. Use a file sander to smoothen the outer edges of the tubing and a sharp tip to remove any flash or shaving from the inner walls.
4. Insert the tube into the nut and ensure that 5/8” enter to the fitting. If fitting is installed near a tube bend, for ¼” tubing there is a 13/16” straight length requirement to allow the tube to be bottomed in the fitting.
5. Tighten the nut by hand and ensure that the threads are not stripped.
6. Use a wrench to perform 1.5 extra rotations to the nut. Do not overtighten as the ferrule will shear the tubing and the fitting will strip.

7. Confirm the connection by unscrewing and screwing again.

For a NTP connection, the procedure is as follows:

1. Secure the component with a clamp (e.g., two way-ball valves with FNPT)
2. Firmly hold the fitting and give five to seven complete turns with Teflon tape.
3. Insert the fitting's MNPT to the valve's FNPT and tighten by hand until finger tight.
4. Use a torque wrench to tighten 8.5-10 lb-ft or turn it 2-3 full turns.
5. Test and ensure that no leaks are present...

Four different union types are present in the apparatus: cross, tee, reducing and straight. As the fluid flow is required to diverge and converge on multiple places, unions connect the tubing and ensure fluid flow through multiple directions.

Figure 18. Fitting components and assembly

3.13 Nitrogen tank

For the back pressure regulator to perform correctly, the thank is required to supply nitrogen gas at a range from 0 to 2500 psi. This pushes the BPR's diaphragm and regulates the fluid output. The tank was anchored to the wall for safety requirements and tubed into the apparatus with fittings, a pressure regulator, and a bleed valve. The pressure regulator is connected straight into the tank and to a 1/4" FNPT to 1/4" tubing compression fitting.

Connected to the reference port is a 1/8” MNPT to 1/4” tubing fitting. A bleed valve is connected to the tubing and located in front of the frame to be reached easily by any user.

Figure 19. Nitrogen tank and regulator

3.14 Frame

The frame was designed specifically to support all the components in the best possible arrangement for the required fluid sequence while ensuring an easy user operation. The total frame dimensions are 5 ft and 10 inches length, 5 feet and 8 inches height and 2 feet wide. Most of the components, excluding the nitrogen tank (for safety regulations) are held by the frame. To allow for future movement, heavy duty rollers are integrated within the frame allowing any user to easily move the entire system and perform testing everywhere. Most of the components are located in the front and with a height range of 3 ft to 6 ft from the floor level for an easy access while operating. Only the overburden pump is located 1 ft above the floor to allow the operator

to pull the lever with easy. Four two-way ball valves are located on the back of the pressure transducers to block/allow fluid for pressure reading.

Accumulators, core holder and pressure transducers are placed vertically to perform adequate testing. All the equipment is expected to stay fixed, and clamps are utilized for every component to limit its movement and always increase the user's safety. However, if required, any component might be removed with a wrench and/or screwdriver. As the laboratory is planned to have a future expansion, the addition of new equipment will still be possible as there is a section of the frame that is empty and can be modified for adding any component for different testing.

3.15 Diamond core Drill

Cores utilized for testing are cut using a M1AA-15 Core Drill from Diamond Products, with an idling speed of 250 rpm, an effective stroke of 20" and a total height of 30". It can be utilized by a regular 110V connection. It included a 1" core drill bit. As the drill must move its position for every core, a special frame was built to attach the drill. Furthermore, to prevent vibrations and curved samples caused from the instability and drill length the core drill is clamped to a 1/2" thick plate with 2" parallel square bars that cover from side to side of the utilized rock.

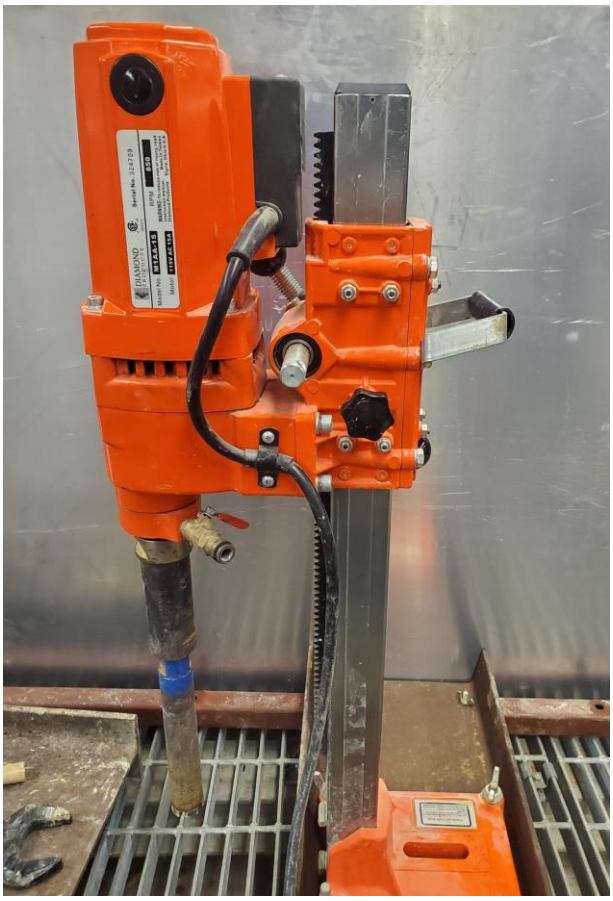
Figure 20. M1AA Diamond core drill

3.16 Data acquisition and electrical connections

3.16.1 Pressure Transducer

The system is composed of three Schneider Electric Foxboro transducers. Two differential and one absolute. Pressure Transducer 1, model IDP10S-T22D11FP-M1L1 has a measuring range from 0-200 psi with a FOXCAL range of 0-300 psi. It is important to denote that despite the range covers up to 300 psi, the transducer will record pressure difference only up to a 200 psi pressure difference. Pressure Transducer 2, model IDP10S-T22E11FP-M1L1 has a measuring range from 0-300 psi. It has a FOXCAL range up to 3000 psi. Despite the display show values up to 3000 psi, record data will stop at 2000 psi difference. Both differential

pressure transducers have a high precision and an accuracy of +/- 0.05% with a response time of 125 milliseconds. Pressure transducers can be calibrated to work at different pressure ranges but have a maximum working pressure of 3626 psi. Exceeding the maximum overrange pressure can cause damage to the transmitter and degrade its performance. Both transducers are limited to a working temperature range from 32 to 140 degrees F. Differential pressure works by measuring two different pressures and calculating the difference. To achieve this, two 1/4" MNPT to 1/4" compression tubing fitting connectors are attached to the High and Low ports of the transducers. The High port is intended to read the pressure on the inlet of the core holder while the Low port is connected to the bottom port of the core holder. It is theoretically known that the entering pressure will always be higher than the exit pressure. If negative values are showed in the LCD display, it is possible that a calibration is required or that the L-H ports are not connected properly. To sense the pressure difference, the pressure transducers utilize a Silicone Oil (DC 200) sensor fill fluid. The process wetted materials (diaphragm or sensor) and the cover are made of 316L Stainless Steel. That pressure difference is showed in an LCD indicator.

Pressure Transducer 3, model IGP10S-TB2F1FP has a measuring range and a FOXCAL range from 0-5000 psi. It has an accuracy of +/- 0.05% and a response time of 100 milliseconds. This pressure transducer measure pressure relative to vacuum. It has a maximum working pressure of 5800 psi. It only has one 1/4" FNPT. A ¼" MNPT to 1/4" compression tubing fitting was utilized to connect the transducer to the center of the core holder to measure the absolute pressure in the center while testing is being performed. It is composed of the same materials as the differential pressure transducers, a silicone oil sensor fill fluid and 316L stainless steel casing [24].

Figure 21. Pressure Transducers

All three pressure transducers have 1/4” NPT ports in the sides for tubing with electrical wire. For electrical connections, the rear cover can be removed by unscrewing the security bolt and rotating the cover counterclockwise. Once the cap is removed, the positive, negative, and ground terminals for power input are in the casing. Furthermore, next to the power ports are the ports for HART communication and calibration. Front cap can be removed in the same way as the rear, removing the cap will lead to the pushbuttons for calibration, range configuration, and multiple transducer operations. The two differential pressure transducers have two-way ball-valves connected to both High and Low ports to avoid any damage to the transducers due to pressure overload. It is recommended that if the pressure is expected to surpass the specified pressure difference or overall pressure limit, to close both High and Low ports for that testing and thus avoid any possible damage. Tubing connected to the transducers is 1/4” stainless steel 316, despite only water and spent acid will reach the transducers, it is highly recommended to flush all the lines with water after the testing has been performed.

3.16.2 National Instruments USB 6002.

To automatically monitor the pressure from the pressure transducers, a Data acquisition system was installed and attached to the frame. It is a National Instruments USB 6002 DAQ device with 4 differential and 8 single ended channels. It has a maximum input voltage of +/-10 Volts and has 2 analog outputs with an output range of +/-10 Volts and an output current drive of +/- 5 mA. It is also suitable for digital I/O operations and serves a +5V Power source with an output voltage of 5 Volts with a +/-3% variation. It delivers a current up to 150 mA. There are three analog input signals connected from the pressure transmitters to the DAQ [25]. However, an expansion plan has been developed and the system is already equipped for future installations. If a heater is to be installed increase the core holder's temperature. A programmed thermocouple will sense the temperature and send voltage signals to the DAQ which will be in charge of turning on/off the relay for the heater's functionality.

3.16.3 LabVIEW software

LabVIEW is the software in charge of recording the data obtained from the pressure transducers. It works by obtaining the instantaneous voltage from the DAQ, scaling it into the PSI scale, filtering the data, converting it into dynamic data, indexing it and displaying it into a waveform chart. DAQ assistant can be manipulated to read multiple samples at a specified rate (Hz). Acquisition mode can be on demand, timed or continuous. It has also a signal input range +/-10 V that can be modified according to the desired voltage range. Once the voltage has been stablished. A custom scale can automatically convert the voltage to the desired range. For this case, as the obtained voltage range from 1 – 5 Volts, for all the pressure transducers, there are two possible methods for scaling the data, a linear correlation with the equation y=mx+b where

the x is the pre-scaled value and y is the scaled value, or a Map range which automatically scales the values proportionally from a range of pre-scaled values to a range of scaled values. For this application, the pressure is not required to be obtained at a high rate. System was configured to read 1 sample at a rate of 0.5 Hz (every 2 seconds). If it is desired, rate can be modified to 1 Hz (every 1 second), 0.25 Hz (every 4 seconds), 0.2 Hz (every 5 seconds), etc. Pressure transducers work by the 4-20 mA current loop. After the it senses the differential/absolute pressure, it sends a bit of current to the DAQ. Since it is only obtaining one bit of information, an RSE terminal communication is configured.

Permeability can be calculated and plotted automatically from the continuous pressure drop data obtained from the transducers. A formula configurator performs the calculations at real time. Darcy's equation is utilized to calculate permeability. Before every experiment is necessary to change the parameters on the front panel to obtain the correct permeability. The parameters are Q = Flow rate (mL/min), L = core length (in), D = core diameter (in), mu = viscosity (cp) and DP = Differential Pressure (psi).

Data is automatically stored after every test with a Write to Measurement File, which creates an LVM file that can be opened from LabVIEW at any time. Data can also be retrieved manually from the generated graphs by right click on the graph and selecting Export data to Excel or Export data to Clipboard.

3.16.4 Electrical connections

Injection pump, refilling pump and pressure transducers require energy to function. For both pumps, they are equipped with a conventional 110V adaptor to plug them to any electrical outlet. However, the DAQ and the pressure transducers are powered with a power supply model

SOLA SDP- 06-24-100T. It has an input voltage range from 85-264 V alternating current (ac) or 90-375 V direct current (dc). The nominal output voltage is 24Vdc and 0.6 A, providing a total power of 15 Watts. It is important to ground all the components properly or noise will be generated. Pressure transducers are required to receive from 12.5 to 42 Vdc while the DAQ accept a maximum of 10 Vdc. For the system to work properly, the components must be connected in series. As mentioned previously, the pressure transmitters use the 4-20 mA current loop principle, this means that when the transmitter senses the minimum absolute or differential pressure (0 psi), a 4mA signal will be generated, on the other hand, a 20-mA signal will be generated at the maximum pressure. For the DAQ to obtain this data, a 250-ohm resistor is added in series to every pressure transducer and DAQ analog channel. Theoretically, once the resistors are in place Ohm's law is applicable. If a 4-mA current is sent from the transducer and with the 250 Ohm resistance, the DAQ will receive 1 Volt as input voltage. On the other hand, the 20-mA current signal along with the resistor will generate a 5 Volt input for the DAQ. However, the actual values vary as the resistors have a 2% error tolerance and the voltage fluctuations might cause minor distortions. To accurately calibrate the transducer's output voltage, release all the pressures until the screen shows a zero value. Use a multimeter to measure the actual voltage on the three pressure transducers. The actual recorded values are 0.998 V, 0.982 V and 0.987 V respectively for the 0-200, 0-2000 and 0-5000 pressure transducers. To record the highest voltage, apply the maximum measuring pressure to every transducer. The recorded voltages are 4.96 V, 4.89 V and 4.93 V respectively for the 0-200, 0-2000 and 0-5000 transducers. Use this information to create a linear correlation and stablish the voltage-pressure scales.

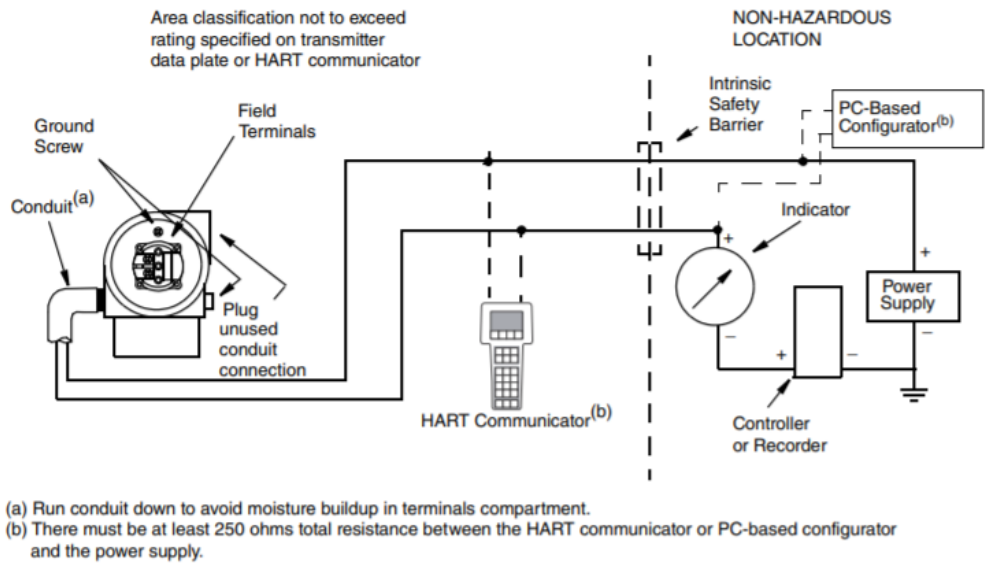

Figure 22. Pressure transducers to DAQ wiring diagram

3.17 Future expansion; Plan and requirements

As the laboratory is expected to be running in a daily basis, future development would involve the addition of a second core holder for bigger core samples suitable for heterogeneity studies and a more precise wormhole distribution analysis. Temperature can also be controlled by the addition of heating system including heating tapes, thermo probe sensors and relays. For a laboratory expansion, a pre-developed schematics has been designed to foresee the components that will be required and their placement within the frame. As it is observed, 8 new two-way ball valves are required to allow fluid to go to either the first, the second or both core holders, they will also be required for controlling overburden and back pressure flow. 5 union tees and 2 union crosses are included as the fluid is required to split its path for reaching both core holders. Another 3 pressure gauges will be placed in the overburden, the entrance, and the exit of the core holder to measure pressure drop. A set of 2 extra three way-ball valves will have the same configuration as in the present core holder, they will control the port for the pressure measurement with the pressure transducers. For all valve connections, 22 male connectors ¼”

MNPT to ¼” compression tubing will be needed. Three ¼” FNPT to ¼” compression fittings for the pressure gauges and approximately 40 ft of tubing. The precise length of tubing will depend on the exact location of the valves. This schematic is suggested for laboratory expansion; however, laboratory can always be modified according to the current research requirements.

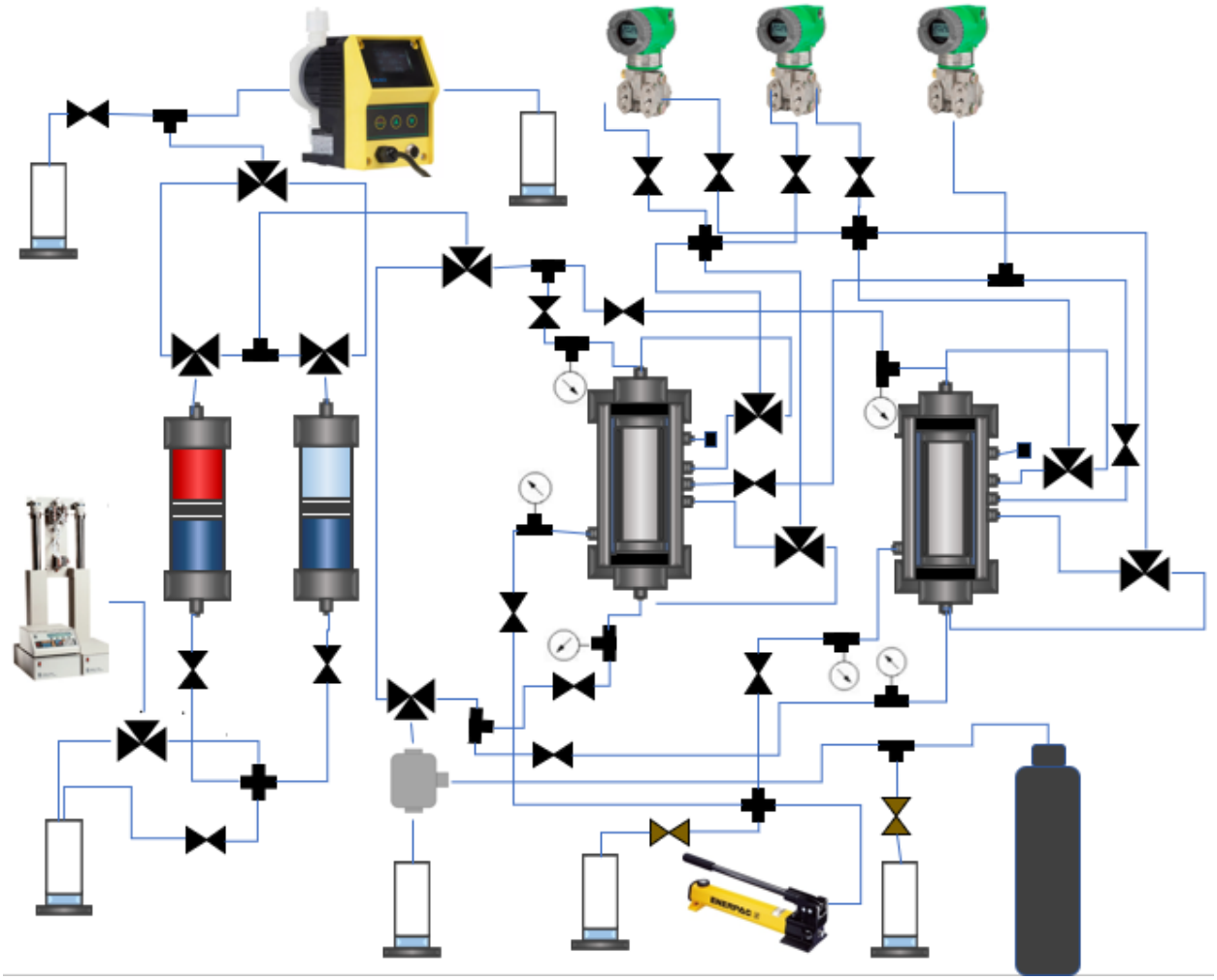

Figure 23. Core flooding expansion schematics

CHAPTER IV

CORE FLOODING APPARATUS OPERATION

To successfully perform a core flooding test, it is necessary to perform multiple chronological steps to obtain accurate and consistent data.

4.1 Safety Precautions

As the laboratory performs at high pressures involving acid, safety should always be prioritized. Every user is required to read the equipment instruction manual and ensure a well understanding of the components and its functionality. Every component, including all valves and equipment are clamped and secured to the frame, thus limiting its movement to zero. However, the user is required to wear the appropriate PPE. It is also recommended for the users to take a laboratory safety precautions risk course. It is required for the user to wear appropriate lab clothes including but not limited to lab coat, safety glasses, face shield, pants/jeans, closed shoes and gloves. Waste disposal container is also available for fluid drop after the test. Every new user is required to be trained and test multiple times with water to get a better understanding of the laboratory operation [26].

4.2 Core drilling

To get the core samples the core drill must be manually placed on top of the rock and secured with heavy duty straps. Connect the water hose and ensure that the water is coming out of the core drill bit. Start the drill and slowly turn the lever to lower the drill. A set of lights on top of the drill show the drill status, proceed applying force is green, lower the force if yellow and remove all pressure if red. Once the core drill bit has reached the desired deepness (6 inches) turn the lever up and extract the sample. If the sample is stuck in the rock, it will be necessary to carefully hit the bottom of the core with a chisel and a hammer. Always ensure to have enough water pressure while drilling to lubricate the cut. If drill is used without water, drill bit will wear at faster rate. Furthermore, it might cause the drill to overheat and damage.

Figure 24. Core drilling procedure

4.2.1 Core preparation (machining)

Once drilled, the cores need to be prepared to perfectly fit into the core holder. As both ends of the core are not flat, it is necessary to turn both sides with the lathe to have a smooth

surface. Measure the sample's length and do not remove more material than the required. Once placed in the lathe do not tight too much as its brittleness will cause it to break. Start the lathe at speeds around 150-300 rpm and remove material at approximately 0.2-0.3 inches per minute. Furthermore, as the drill must be moved for every core, it is not ensured that it will be completely rigid every time, hence, the vibrations and movement will lead to a curved sample. In case of a sample with curved walls, it is recommended to use the sander machine to smoothen the surface and have a straight sample.

4.2.2 Core saturation and porosity measurement

Understanding the structure, heterogeneity, and porosity of the sample to be tested is crucial for data analysis. There are many methods for obtaining this information such as a Computer Tomography scanning to obtain a 3D visualization of the rock heterogeneity including vugs, pores and fractures. Scanning the sample before and after the test helps to visualize the matrix acidizing and wormhole formation procedure [27].

Porosity is defined as the percentage of void space in a rock, in other words, is the amount of space in the rock, and is calculated by dividing the pore volume over the bulk volume. Bulk volume is simply obtained by applying the cylinder's volume equation while pore volume can be calculated saturating the core. The method to calculate pore volume is by core saturation. This involves the core being placed in a saturation cell (vacuum chamber) full of clean water for 24 hours. The vacuum will cause the air in the pores to exit the rock and the water will periodically fill the spaces. To perform the desired calculation, measure its weight before and after saturation. The difference from the dry and wet rock masses will determine the pore volume. It is crucial to measure height and length of the sample to obtain the volume of the sample.

To obtain porosity, equation 1 is applied:

$$\phi = \frac{PV}{BV} = \frac{Wet\ rock\ mass - Dry\ rock\ mass}{D^2 * \frac{\pi}{4} * L} * 100 \quad (1)$$

The units for the dimensions are Mass in grams (g), diameter in centimeters (cm), length in centimeters (cm).

4.3 Laboratory inspection and equipment startup

Before proceeding to any operation, review the safety requirements and wear the proper equipment for testing with acid. Once all the safety guidelines have been fulfilled, it is necessary to spend time checking all the tubing connections and ensure that the fittings and components are in the correct configuration. Injection pump should be full, overburden pressure pump oil level should also be revised and ensure that the nitrogen does not present any leak. Take the necessary time to study and understand the valve configuration

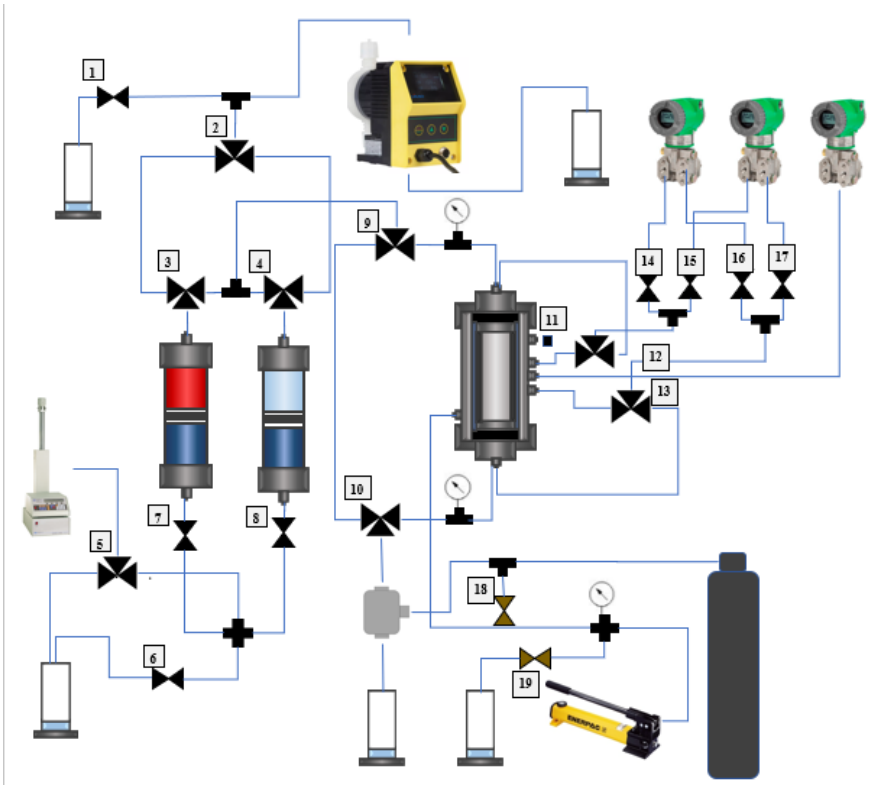

Figure 25. Valve labeling and position

Table 5. Valve specifications and location

Valve #	Valve location	Type	Port size	Material	Temp. F	Pressure	Supplier
1	Refilling pump bypass	2-way	1/4" FNPT	SS 316	350	6000	Superior Fluids
2	Refilling pump fluid divider	3-way	1/4" FNPT	SS 316	350	6000	Superior Fluids
3	Acid accumulator inlet/outlet (top)	3-way	1/4" FNPT	SS 316	350	6000	Superior Fluids
4	Brine accumulator inlet/outlet (top)	3-way	1/4" FNPT	SS 316	350	6000	Superior Fluids
5	Syringe pump refill/injection	3-way	1/4" FNPT	Cast Iron	185	7250	TOOLOTS
6	Injection hydraulic oil discharge	2-way	1/4" FNPT	Cast Iron	185	7250	TOOLOTS
7	Acid accumulator oil inlet/outlet	2-way	1/4" FNPT	Cast Iron	185	7250	TOOLOTS
8	Brine accumulator oil inlet/outlet	2-way	1/4" FNPT	Cast Iron	185	7250	TOOLOTS
9	Core holder inlet / bypass line (top)	3-way	1/4" FNPT	SS 316	350	6000	Superior Fluids

10	Core holder outlet / bypass line (bottom)	3-way	1/4" FNPT	SS 316	350	6000	Superior Fluids
11	Overburden relief	Relief	1/4" FNPT	SS 316	350	6000	Superior Fluids
12	Core holder to pressure transducer (top)	3-way	1/4" FNPT	Cast Iron	185	7250	TOOLOTS
13	Core holder to pressure transducer (bottom)	3-way	1/4" FNPT	Cast Iron	185	7250	TOOLOTS
14	Pressure Transducer 0-300 psi Low port	2-way	1/4" FNPT	Cast Iron	185	7250	TOOLOTS
15	Pressure Transducer 0-300 psi High port	2-way	1/4" FNPT	Cast Iron	185	7250	TOOLOTS
16	Pressure Transducer 0-3000 psi Low port	2-way	1/4" FNPT	Cast Iron	185	7250	TOOLOTS
17	Pressure Transducer 0-3000 psi High port	2-way	1/4" FNPT	Cast Iron	185	7250	TOOLOTS
18	Nitrogen discharge	Needle	1/4" FNPT	SS 316	350	6000	Swagelok
19	Overburden oil discharge	Needle	1/4" FNPT	SS 316	350	6000	Swagelok

4.3.1 Syringe pump configuration and parameters.

The pump consists of two components, the pump, and the controller, ensure that the electrical power outlet is fully connected and turn on the pump buy pressing the red buttons on the front of the pump and the controller. Once started, follow the instructions below:

1. Check the valve configuration for injection pump refilling.
2. Hydraulic oil beaker should have a minimum of 300 mL if the pump is completely empty. Otherwise, it will absorb air and performance will not be accurate.
3. Press the “refill” blue button and wait for the pump to fill with oil.
4. Once the pump is full, screen will show 266.43 mL
5. Select the desired parameters for fluid injection

6. For constant flow select “const flow” blue button. Then press “1”, enter the desired flow and press “enter”.
7. For constant pressure press the “const press” blue button. Then press “1” and enter the desired injection pressure and then press “enter”.
8. Pump limits can be stablished by selecting “limits” blue button.

4.4 Core mounting into the core holder

The core holder assembly is designed to always stay fixed in the frame, however, if maintenance is required, an Allen-key is provided to remove the core holder and proceed the core holder maintenance section. Core holder supports a 1” x 6” core sample, with the use of spacers, length can significantly vary according to the requirements. This laboratory permits a core down to 1” x 4” with the aid of a 2” spacer. If desired, multiple spacers can be machined or purchased from the manufacturer. For loading the core holder proceed as follows:

1. Unscrew the top stationary top cap to release the slider’s exerted pressure.
2. Locate the bottom radial knob on the bottom threaded cap and untight to enable the bottom slider’s rotation.
3. Slowly rotate the bottom slider until the tabs align and the bottom slider can be extracted from the core holder.
4. Insert the core holder from the bottom of the core holder and place the bottom slider back in position, rotate until the tabs overlap.
5. Tighten the radial knob to lock the bottom slider and prevent twisting.

6. Place the top stationary cap back in position and tighten until both slides compress the sample and no movement is allowed. This ensures that the core sample is in position and in full contact with both sliders

4.4.1 Core holder components and maintenance

The core holder consists of multiple components that allow its functionality. The main cylinder, which is the one in which all the components assemble. Screwed to the cylinder are the top and bottom caps, that allow the cylinder to seal and hold the Viton sleeve as well as the top/bottom cap components. Attached to the top/bottom caps, a smaller plug of 1” diameter containing both 1/8” tubes slide into the caps and allows the core holder to seal from the top and the bottom. The Viton sleeve is a rubber cylindrical body that encloses the core sample from the sides and pressurizes the walls as overburden pressure is applied. Finally, a blue core holder frame assembled to the main frame that holds the core holder into a fixed position.
Maintenance is recommended every 10 experiments as hydrochloric acid and the stagnant fluid might corrode, oxidize and deteriorate the system. Another common problem is the Viton sleeves shatter, this occurs when the core holder is not properly sealed, and the top/bottom sliders are not fully in contact with the sample. When overburden pressure is applied Viton sleeve gets damaged and cracks form allowing hydraulic oil to get into the core sample and exiting the core holder from the bottom slider. For a Viton sleeve replacement and maintenance, the procedure is as follows:

1. Unscrew the four top and bottom tubing connectors.
2. Open the hydraulic oil bleed valve to release the hydraulic oil stuck in the core holder.

3. Unscrew the three pressure transducer connectors and the two overburden connections from the side of the core holder.
4. Remove the top and bottom sliders along with the spacers.
5. With the proper Allen key, remove the hex bolts from the blue core holder frame.
6. Place the core holder into a safe place and in horizontal position.
7. Double check and release any hydraulic oil left over.
8. Unscrew the top and bottom caps.
9. Use the seal grip remover tool to remove the top and bottom cylinder seals from the core holder.
10. Remove the used Viton sleeve and place the new one.
11. Use the insertion grip tool to place the top and bottom cylinder seals back to the core holder.
12. Repeat backwards steps 8 to 1.

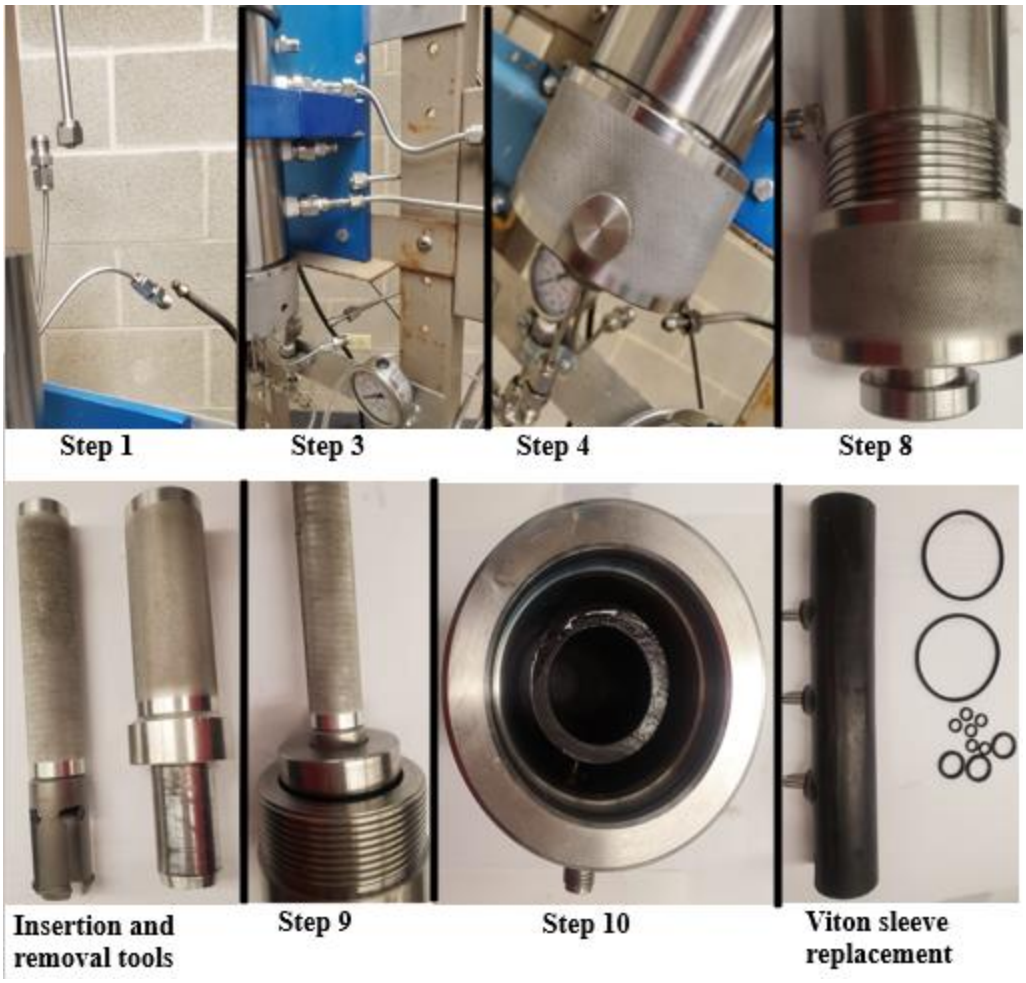

Figure 26. Core holder maintenance procedure

4.5 Refilling system

A diaphragm pump is utilized for refilling the accumulators with water and acid. Before starting the pump ensure that it is correctly connected to the electrical outlet. The pump can be configured to work at multiple speeds; however, it is recommended to use 100% speed as its capacity is 1L/hr. The accumulators are 275 mL, which means that it will pump approximately 16 ml/min, or a total of 16.5 minutes will be required to refill the entire accumulator. To refill the accumulators:

1. Pour the desired volume of water/brine into the beaker. It is recommended to add extra 100 mL of fluid to the beaker to ensure that the metering pump will not absorb air.
2. Ensure the beaker is placed correctly in the stand and that the clear hose is reaching the bottom of the flask.
3. Check the valve configuration for acid/brine refilling.
4. Ensure that the accumulator is full of hydraulic oil and that the piston has reached the top.
5. If fluid is left on the accumulator, use the syringe pump to inject hydraulic oil into both accumulators and push the piston to the top end.
6. Pistons have reached the top end once the syringe pump starts building pressure and no water is coming from the fluid outlet. Stop the pump and release the injection pressure (check syringe pump valve configuration for correct procedure).
7. Press the start button on the metering pump and it should immediately start pumping the brine/acid into the respective accumulator.
8. If the valves are positioned correctly, hydraulic oil will immediately start exiting by the oil injection exit line.
9. Once the desired volume of fluid has been reached press the stop button.
10. Remove the flask from the base, dispose the remaining fluid in the waste disposal unit and carefully clean it with water.
11. Fill the flask with clean water and open the refilling exit valve.
12. Turn on the pump and let it run for at least 5-8 minutes to clean the pump from any acid residue.

4.5.1 Accumulator air release procedure

If inconsistent data is being obtained because of pressure disturbances or bubbles come out of the tubing while testing, it is possible the accumulators have been filled with air, if the user suspects so, it is important to stop any experiment from being performed and proceed to the following instructions:

1. Release the pressures from overburden pump, injection pump and backpressure
2. Open the bypass line to let all the brine/acid exit the system.
3. Refill the syringe pump and inject hydraulic oil into both accumulators until a significant pressure increase is observed (around 1,000 psi).
4. Stop the syringe pump and open the syringe pump two-way ball valve to release the pressure built and to allow the hydraulic oil to exit the system.
5. With the metering pump, inject 275 mL of water to the first accumulator until it is full.
6. Repeat step five for the second accumulator.
7. Repeat steps 3 to 6 one more time to ensure that there is not air leftover. Bubbles should come out of the tubing after the fluid stops denoting there was air present in the accumulator.

4.6 Apply Overburden pressure

Overburden pressure is applied to confine and compress the sample in all directions normal to the sample's walls. Pressure should always be at least 300 psi above the injection pressure. For overburden pressure, the steps are as follows:

1. Open the vent cap on top of the pump and check the hydraulic oil level. If necessary, add the required amount of ENERPAC HF blue oil.
2. Close the cap and check the knob on the front right side. If open rotate clockwise to close it.
3. Move the lever up and down a few times until the system is full of oil.
4. Once the pressure builds up, check the overburden pressure gauge and pump approximately 300 psi Do not add more than 500 psi initially as the core might break or damage.
5. Look for any leak or system malfunction. If a leak is detected. Open the overburden pressure needle valve to release the hydraulic oil from the system.
6. If system is working correctly maintain 300-500 psi above the injection pressure the entire experiment to prevent the fluid from going through the sides of the core sample. Correct overburden application will ensure the fluid's injection through the center of the core.

4.7 Apply back pressure

Back pressure is applied to simulate downhole conditions and to maintain the carbon dioxide in aqueous solution. Nitrogen tank is connected to a multi-stage regulator that can reduce the inlet pressure by 90% on the first stage. The second stage is a knob that increase/decrease the outlet nitrogen pressure. The nitrogen tank should always remain closed and only open when testing will be performed. To open the nitrogen tank, turn the top gray knob counterclockwise. The Airgas regulator will show an increase of 2500 psi (right gauge) and the left gauge will show the actual exit pressure. To fully open the tank outlet, turn the black knob on the rear of the

regulator. Rotating the front knob with the label AIRGAS will increase/decrease the nitrogen outlet. The outlet pressure will be displayed in the left gauge.

4.8 Pressure transducers

Before proceeding, check that the pressure transducers readings are zero. If not, open the discharge ports located on the front of the pressure transducers. For the 0-200 Pressure transducer bolts are labeled as “07B”, for the 0-2000 is labeled “06C” and “08B” for the absolute pressure transducer. Open both High and Low ports by slowly rotating the bolt counterclockwise. Once the pressure display is zero, close the bolts and start the operation. If pressure is still not zero, proceed to the Pressure transducer calibration section. If test pressure is expected to surpass the specified limits for the differential transducers, close the to way-ball valves for the respective transduce. Valves are located on the rear part of the frame.

Pressure transducers can be calibrated for different ranges. Despite they come factory calibrated, if temperature is changed or pressure shows a value being non-zero after multiple tests, is suggested to re-zero it for better outcome. Pressure range is expressed with two terms: lower range limit (LRL) and upper range limit (URL). If no range is configured, LRL will stay as zero and URL being the upper range limit, in this case 200 and 2000 psi for both differential transducers. Lower and upper values can be calibrated by applying the desired pressure and selecting either CAL LRV or CAL LRV. It can also be programmed without pressure by entering the new database manually. Zeroing the transmitter is one of the most important steps as periodically it starts to show small values and calibration is required. To perform this procedure, proceed as follows:

1. Locate the front discharge ports and use a wrench to untight the bolt. This will allow all trapped fluid to exit the transducer and have an accurate calibration.
2. Locate a flat head screwdriver and unscrew the small bolt located on the front and preventing the cap from rotating.
3. Rotate the cap counterclockwise and remove it from the transducer.
4. Select “Next” multiple times until “CALIB” word appears. Select “Enter”
5. Select “Next” until “CAL AT0) is displayed.
6. Select “Enter” and wait for the transducer to calibrate.
7. Select “Next” until the word “Save” appears. Finally select “Enter”
8. Pressure reading should be 0 psi.
9. Place the front cap and the security bolt back in place.

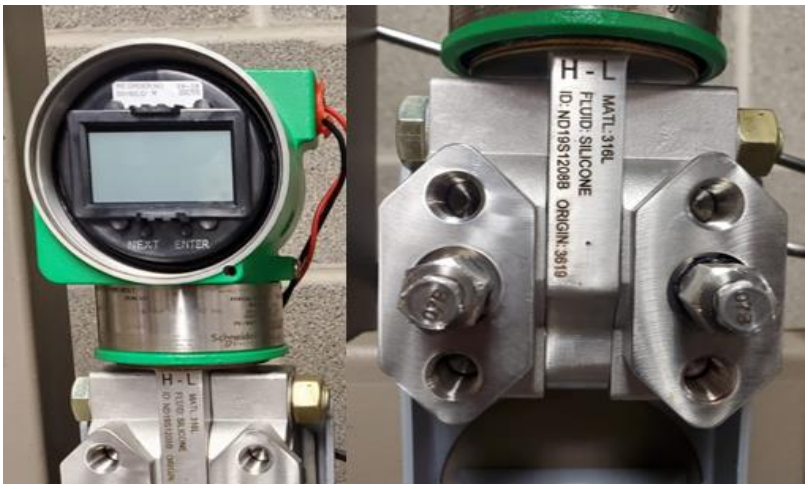

Figure 27. Pressure transducer calibration

4.9 Open LabVIEW

LabVIEW program is crucial for data recording. Before starting the injection, open the “Matrix acidizing” file. Front panel and block diagram should be displayed. Only if sample rate and number of samples obtained will be changed, navigate through the DAQ assistant in the

block diagram and modify to the desired settings. Select the front panel window and modify the “L”, “Q”, “mu” and “D” parameters to the actual sample measurements. Waveform charts can be modified to show different appearance, display format, plots, and scales. Once ready, hit the small arrow button with the “run” title.

4.10 Start Brine injection

Once LabVIEW starts recording data, the syringe pump can start injecting brine. The following steps are as follows:

1. Check valve configuration for brine injection to core holder.
2. If constant flow is desired, enter the flow rate and press “run” blue button. The timer should immediately start, and the pump’s fluid should start decreasing.
3. Pressure transducers will start displaying values and four different LabVIEW graphs should be running (3 for pressure transducers and 1 for permeability).
4. Pressure will build up until it reaches steady state
5. Once the pressure drop becomes constant the permeability chart will show a horizontal straight line with a 2-3 psi tolerance. Permeability can also be calculated manually to confirm that matches the LabVIEW data.

4.11 Start Acid injection

Once the steady state has been constant for a few minutes, the following steps are performed:

1. Open the Hydrochloric acid accumulator and close the brine accumulator.
2. Immediately record the time and the remaining pump volume.

3. According to the calculations performed for the piping volume and the flow rate, estimate the time required for the acid to reach the core.
4. Once the acid reaches the core, pressure drop will start decreasing as the acid will be reacting and wormholes will appear.
5. Once the pressure drop becomes zero or close, the acid has successfully created a breakthrough.
6. Record the time and the volume of fluid remaining on the injection pump.

4.12 Procedure after matrix acidizing

After the acid breakthrough, the system is required to post-flush and depressurize. Injection pressure, backpressure and overburden pressure should be released after removing the acid from the core holder.

1. . After the acid breakthrough, press “stop” in the injection pump.
2. Go to Front panel in LabVIEW and press the “stop” button.
3. Close the valve for the acid accumulator.
4. Close the nitrogen back pressure by turning the top gray knob clockwise.
5. Slowly open the Nitrogen gas needle valve, the gas should dissipate in a few seconds.
6. Close the small black knob on the back of the nitrogen tank by rotating it clockwise.
7. Release overburden pressure by opening the needle valve. Do not open the valve entirely. At least 100-200 psi of overburden pressure should remain in the core holder. If all overburden pressure is relieved, spent acid and brine will come out from the bottom of the core holder and can potentially cause an equipment damage or put in danger the user’s safety.

8. Fill the refilling flask with clean water. Turn on the metering pump and use the refilling brine valve configuration to refill the brine accumulator with water.
9. Use the refilling acid valve configuration to refill the acid accumulator with clean water.
10. Once both accumulators are full, use the syringe pump refilling valve configuration to refill the syringe pump.
11. Open both accumulator valves and use the syringe pump to flush all the system until both accumulators empty.
12. Repeat steps 8 to 11 to ensure that acid residuals are completely flushed.
13. Stop the injection pump and release the pressure by opening the hydraulic oil discharge valve.
14. Release the remaining overburden pressure by opening the needle valve.
15. Use an adjustable wrench to untight the two bottom core holder connections (pressure transducer and BPR connections).
16. Untight the small knob and slowly remove the bottom plug insert.
17. The core sample should slowly slide through the bottom of the core holder.
18. Place the bottom plug insert back and connect the tubing to the accumulator.
19. Clean the sample and analyze the obtained data.

CHAPTER V

RESULTS

To ensure correct lab setup and functionality, a set of experiments were conducted with low permeability limestone, non-carbonate rock, and high permeability limestone. These results show the reliability and the consistency of the obtained results. Parameters such as porosity, permeability tubing volume, pressure drop across the tubing are calculated with the data obtained from the experimental results.

Before and after performing any test, core sample measurement procedure should be performed, this will help with the porosity and permeability calculations. Before any experiment it is recommended to measure length, diameter, dry and wet mass. Labeling the core is also important to keep a clean record of the measurements and core lithology. Once core flooding has started, to reduce the useless data generated from the graphs, a calculation of the time that the fluid takes to reach the core sample will be helpful to predict the exact time when the acid reaches the sample. If constant pressure is selected, monitor, and record the flow rate as it will change to maintain the pressure. If constant flow rate is selected, record the injection pressure.

Brine will flow first until steady state has been reached. Once reached, record the time and volume of oil in the syringe pump. Open the acid valve and immediately record the time and volume again. Estimate the time that will take the acid to reach the core and record the remaining

volume in the pump. Finally, once the pressure drop reaches zero, record the time and the volume in the injection pump. Fluid volume in the spent acid/brine beaker should match the volume spent by the injection pump.

Once all the information has been recorded, a set of equations for porosity and permeability can be automatically calculated by LabVIEW, manually or with excel software. Once the brine injection has reached steady state and the core is fully saturated, permeability can be calculated using the core dimensions and the differential pressure transducer's data. To calculate permeability equation 2 is utilized:

$$k = \frac{96.43 * 4 * Q * L * \mu}{\pi * D^2 * \Delta P} \quad (2)$$

where flow rate (Q) is in mL/min, core length (L) in inches, viscosity (μ) in cP, diameter (D) in inches and Pressure drop (ΔP) in psi. Permeability is then utilized to calculate the pore volume to breakthrough, in other words, the volume required for the acid to create the wormhole. This information is useful as it helps to calculate the optimum injection rate for that lithology. To accurately obtain information, perform multiple experiments using different flow rates. To calculate the pore volume equation 3 is utilized:

$$PV = \phi * \frac{\pi}{4} * D^2 * L \quad (3)$$

where diameter (D) and length (L) are in centimeters.

As previously mentioned, volume calculation for tubing is directly proportional to the time that is takes for the acid to travel from the accumulator to the core. For tubing volume calculation:

$$V = L * \frac{\pi}{4} * (D_i)^2 \quad (4)$$

where volume (V) is in cm^3, pipe length (L) is in centimeters, and tube's inner diameter is in centimeters.

Having calculated the tubing volume from the accumulator to the core holder's inlet. The following equation is used to obtain the time for the acid/brine to reach the core sample.

$$t_{fluid} = \frac{V}{Q} \quad (5)$$

where $t_{fluid}$is in seconds, volume (V) is in cm^3 and flow rate (Q) is in mL/min.

For the Indiana limestone tested sample, previous parameters can be calculated, a 10 mL/min flow rate and no back pressure test will be utilized, results are as follows:

- Porosity: $\phi = \frac{PV}{BV} = \frac{41.719-41.488}{(1*2.54)^2*\frac{\pi}{4}*(1.448*2.54)} \times 100 = 3.14\%$
- Permeability: $k = \frac{96.43*4*10*1.448*1}{\pi*(1)^2*(12)} = 148.23\ mD$
- Pore volume: $PV = 3.14\% * \frac{\pi}{4} * (1 * 2.54)^2 * (1.448 * 2.54) = 0.584 cm^3$
- Volume of tubing: $V = (41 * 2.54) * \frac{\pi}{4} * (0.12 * 2.54)^2 = 7.59 cm^3$
- Time to reach core: $t_{fluid} = \frac{7.59\ mL}{5 mL/min} = 1.52\ min$

5.1 Low permeability Limestone

Matrix acidizing is not suitable for all types of carbonate reservoirs. Low permeability reservoirs do not offer the ability for the acid to flow and thus, no wormhole ramifications are

developed. A test was performed to evaluate the laboratory behavior and the acid/rock interaction.

A core sample with dimensions Diameter=0.964” and Length=5.82” was tested using 150 mL of 15% HCl. Back pressure was set to 800 psi. Flow rate was set to 5 mL/min and overburden was maintained 300 psi above the injection pressure the for the entire experiment. Initial weight of the sample was 177.17 g.

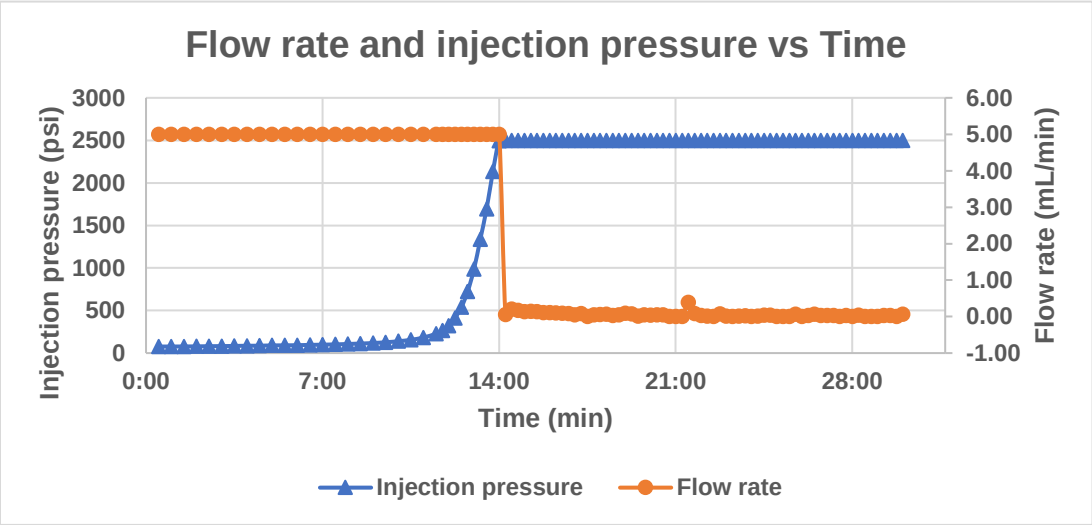

Figure 28.Flow rate and injection pressure vs Time

Maximum injection pressure was set to 2500 psi as it is still considered a safe injection pressure and pressure transducers do not support further injection pressure. As the tubing was completely empty and the flow rate was 5 mL/min, figure 28 shows thar the time required for the fluid to fill the tubing, the core and build pressure was around the 10:30 minutes. It took 3:30 minutes to go from 156 to 2500 psi. Once the pump reached its programmed maximum pressure, flow rate decreased to 0 mL/min. Calculated permeability is 0.35 mD, however, this permeability is assuming that there was a flow rate of 5 mL/min at 2500 psi. As there was no fluid flow, this

permeability is not applicable, however, it is helpful to assume that it is less than the calculated. For permeabilities less than 1 mD it is recommended to use hydraulic fracturing.

Figure 29. Acid injection in low permeability limestone

For a better understating in the acid/rock interaction behavior, a small sample was completely submerged in HCl for a designated time and the weight was measured along with its dimensions.

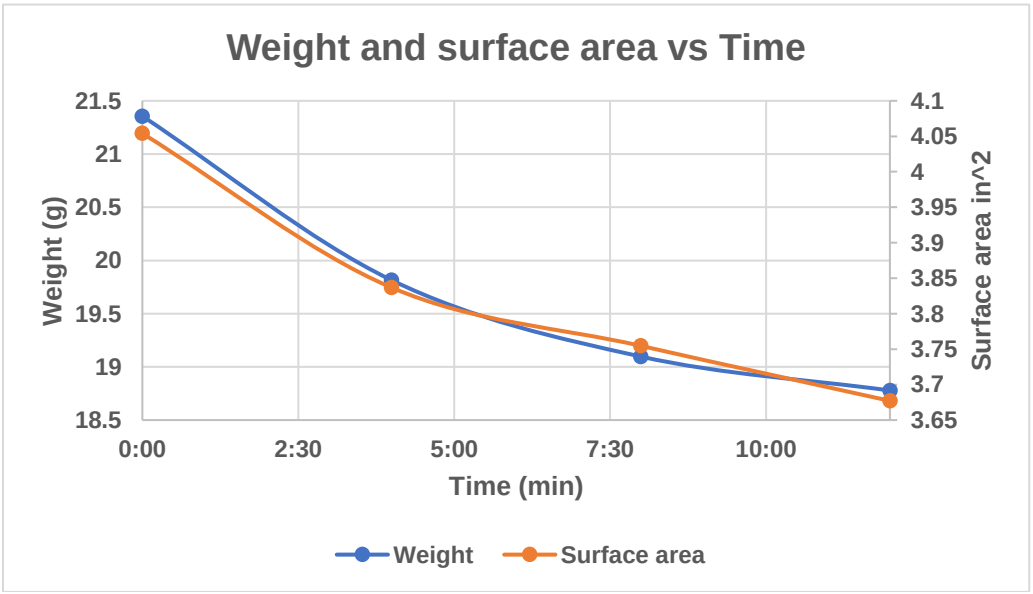

Figure 30. Weight and surface area vs time

Initial surface area was 4.0541 cm^2 and a weight of 21.356. Sample was submerged in 50 mL of 15% HCl solution. Sample was taken out after 4, 8 and 12 minutes. Surface area after 12 minutes was 3.6771 cm^2 and weight was 18.778 grams. As previously explained in chapter 2, it is a first order exothermic reaction where carbon dioxide gas, water and calcium chloride are produced. In high permeability reservoirs, this consumption rate and weight reduction is caused by the wormhole creation. As this limestone does not present a good permeability Injected acid would agglomerate and react on the sample's top surface No diversion was observed, however it consumed the top surface and created a top double-curved surface with a 0.022" of material removed from the initial center point to the after acidizing center point.

5.2 Non-carbonate sample with low permeability

A test was performed on a lava rock to observe the laboratory behavior. Knowing the type of formation is crucial to understand its composition (minerals present) and successfully perform an acid diversion. Carbonate formations are mostly acidized with HCl as it will dissolve the carbonate-based materials to create the channels or wormholes and allow the formation fluid to flow. This is considered a simple reaction as it occurs in a single stage. HCl reacts with carbonate rock and creates water, carbon dioxide and salt. Sandstone acidizing is considered a more complex reaction as Hydrofluoric acid is mixed with Hydrochloric acid to create what is known as "mud acid". This combination dissolves the minerals present in the sandstone such as quartz, clay and feldspar causing a three-stage reaction. Matrix acidizing on volcanic rock was performed to study and evaluate the system's performance along with the core sample behavior.

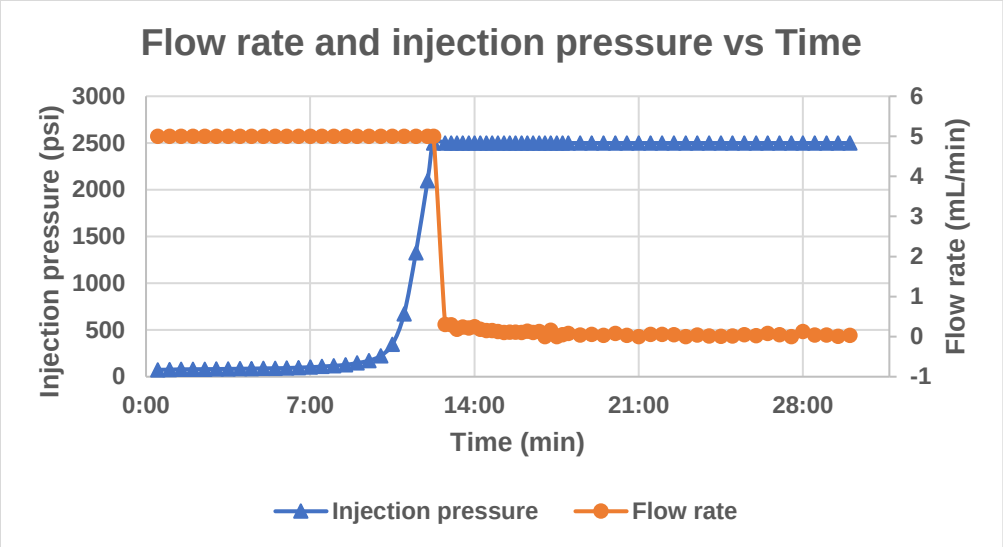

Figure 31. Injection pressure and flow rate vs time

The observed behavior is similar to the low permeability limestone. Injection rate was set to 5 mL/min. Applied overburden pressure was kept 300 above the injection pressure for the entire experiment. Core dimensions are 3.045” length and 0.972” diameter. Overburden pressure was set at 300 psi. It took approximately 10 minutes for the fluid to reach the core, however, once reached, no permeability was observed. Because of that, there was an abrupt increase in injection pressure to maintain the flow rate.

Both parameters are inversely proportional, a sudden increase in injection pressure yields a sudden pressure drop. As the core presents an almost zero permeability, regardless of the injection pressure no diversion will be observed. Maximum injection pressure of 2500 psi was maintained for 17 minutes with no change in parameters.

Figure 32. Non-carbonate sample

Volcanic rock is commonly found in the subfloor, however, the utilized technique for this type of rock is hydraulic fracturing. Due to its composition with high amounts of iron and magnesium they do not present a high acid interaction as limestones or sandstones. Despite its large pore volume percentage, permeability is close to zero as the pores are not connected and fluid cannot pass through.

5.3 Thru-hole core sample

One sample with a drilled thru hole was acidized to evaluate the conciseness of the data obtained under different conditions as well as the acid behavior inside the core holder. The sample was machined in the lather and a 1/8” long drill bit was used to drill throughout the specimen. In matrix acidizing, once the wormhole has been formed, pressure drop is expected to reach any value from 0 to 1 psi as the inlet and outlet pressures become almost equal. The main purpose of the test was to simulate a scenario where the wormhole has been created and the pressure drop across the core.

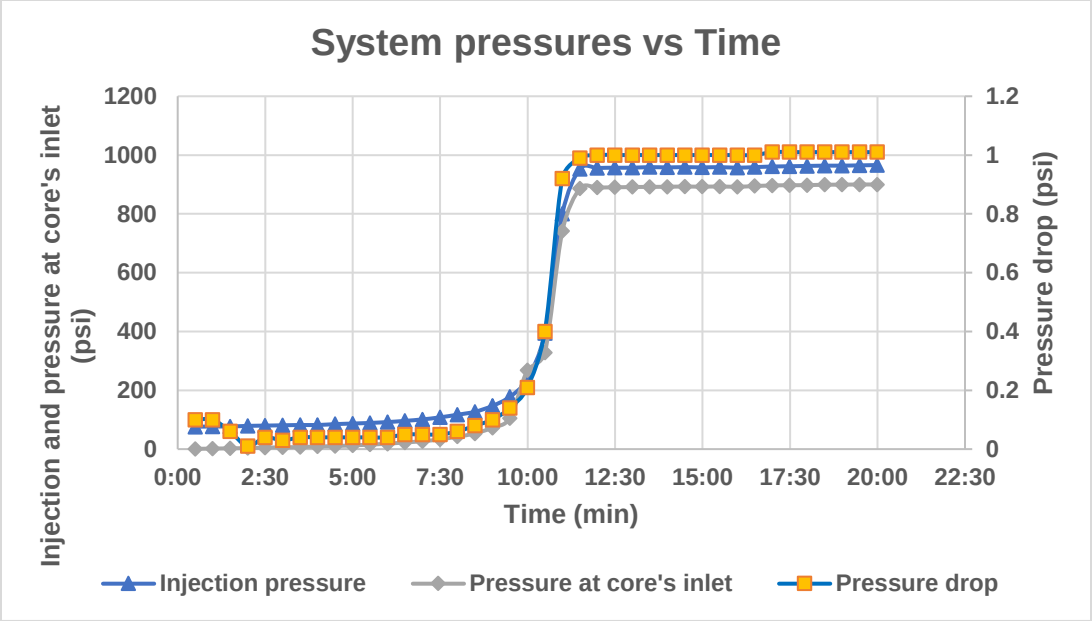

Figure 33. Pressures around the core vs time

The sample tested had a length of 5.804”, a diameter of 0.969” and 171.402 grams dry weight. A thru-hole was drilled in the middle of the sample. It had a 0.188” diameter. 800 psi of back pressure were applied, and overburden pressure was kept 300 psi above the injection pressure the entire experiment. Injection flow rate was set to 8 mL/min and 200 mL of acid passed through the sample. Test results show that it took approximately 10 minutes for the lines to fill, and pressure started its increase. An average of 961 psi injection pressure were required to maintain the 8 mL/min flow rate requirement through the sample with the inputted back pressure.

Figure 34. Thru-hole sample before and after acidizing

Test was stopped after approximately 23 minutes as the HCl finished. Figure 34 shows that there was an increase in the hole diameter from 0.188” to 0.224” in diameter. Final sample weight after the core flooding was 169.594 grams.

5.4 Overburden pressure test

To test overburden pressure, it was required to perform multiple system modifications. For this test, pressure was monitored utilizing pressure transducer connected to the overburden port and another connected to the inlet port of the core holder to measure the actual inlet pressure. System was first tested without overburden pressure to observe the behavior of the pressure drop and the fluid’s traveling path. Flow rate was set to 5 mL/min and there was no back pressure applied.

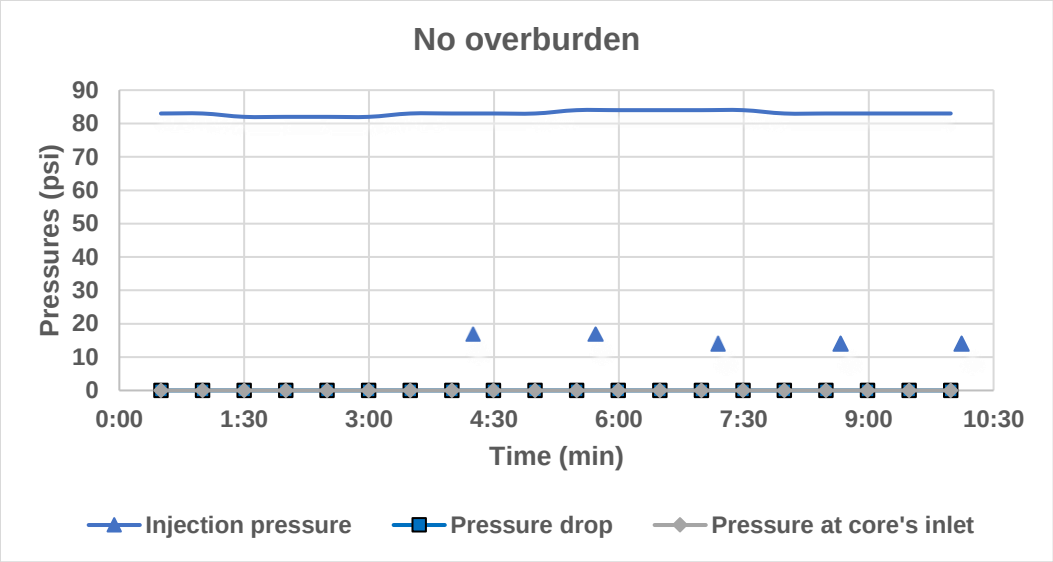

Figure 35. Overburden test pressure vs time

As expected, the pressure did not build up the entire test, injection pressure maintained constant between 82-85 psi. Pressure from the inlet of the core was also measured the entire time. Test was stopped after 10 minutes as the pressure drop was 0 psi the entire test. Remaining pump's hydraulic oil was 215.68 mL with a total of 50.7 mL consumed during the 10-minute test. Diversion was observed after 2 minutes; however, fluid was no exiting through the exit port, on the other hand, it was exiting through the bottom part of the core holder. Fluid exiting through the core holder was measured for 8 minutes, and the total count was 35 mL. This volume was a proximate since the fluid would spill towards multiple directions due to the flow through the bottom of the core holder's slider and the tubing. This fluid behavior was caused because of the lack of overburden pressure. Fluid enters the core holder and immediately contacts the sample, since there is no back pressure and/or overburden pressure, fluid travels to the side walls, flowing between the Viton sleeve and the core sample. Finally, as no pressure keeps it within the sample walls, it exits through the bottom of the core holder, skipping the slider and neglecting the exit's port.

To confirm the analysis performed previously, a second test was executed with the same 5 mL/min flow rate, but this time, overburden and back pressure were applied. Back pressure was set to 400 psi and overburden was maintained approximately 300 psi above the pressure entering the core. Experiment ran for 30 minutes, and permeability could also be calculated.

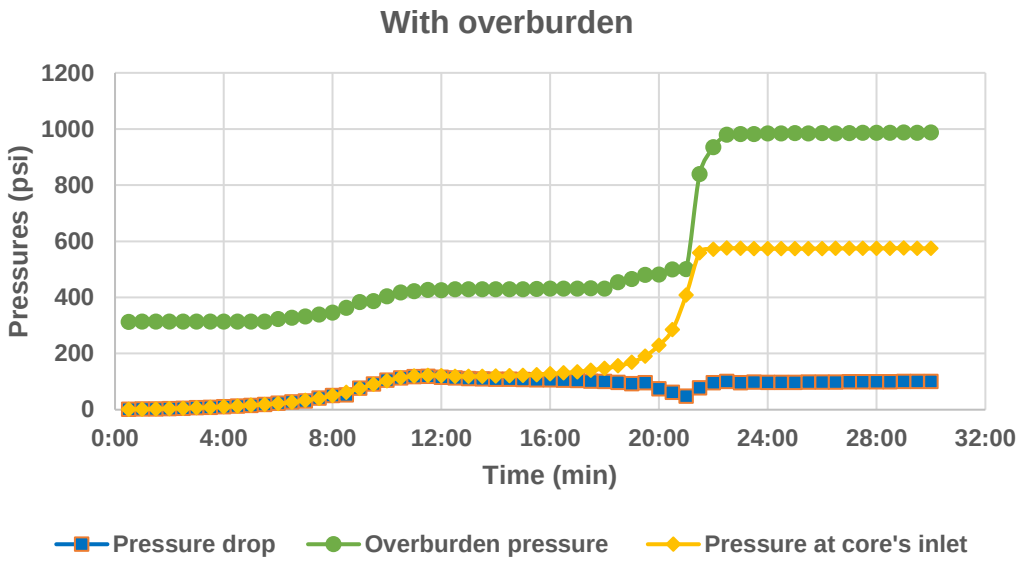

Figure 36. Overburden test 5 mL/min pressure vs time

Three pressures were plotted to show the correlation between pressure drop, pressure at core's inlet and overburden pressure. It took 20.5 minutes build the pressure and have diversion. Exiting fluid volume was measured for 8.5 minutes and a volume of 44 mL was gathered from the exit beaker. After diversion occurred, the pressures stabilized and maintained constant for the rest of the experiment. The maximum injection pressure was 647 mL, the maximum pressure recorded at the core's inlet was 575.4. As overburden pressure is constantly maintained 300 psi above the inlet's pressure, fluid can not travel through the Viton sleeve as with no overburden. It is forced to pass through the sample and thus an average pressure difference of 98 psi is required to have fluid flow.

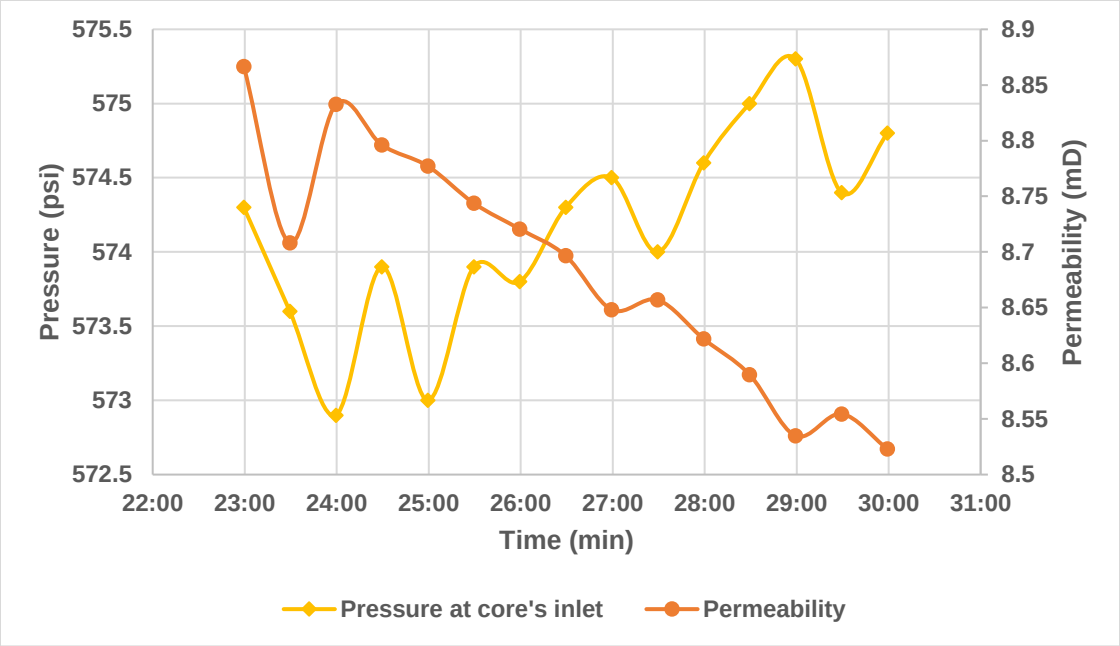

Figure 37. Overburden test, permeability vs time

A permeability plot complements the statement by exposing the steady state achievements after 23 minutes. From 19.5 to 22 minutes a sudden pressure increase occurred, that is exactly when it increased significantly from 268 to 643 psi and diversion occurred.

5.5 Pressure drop across the lines

Core flooding apparatus was compactly designed to avoid fluid and pressure loss within the tubing. A test to calculate the pressure drop from the accumulator outlet to the end of the line is helpful to determine and analyze the injected pressure by the pump vs the actual pressure entering the core when acidizing. If the Syringe pump's data is used to analyze the wormhole creation, calculations will be erroneous. Results obtained from this test should match the results obtained by the differential pressure transmitters and thus proving the amount of pressure loss by the length of the tubing.

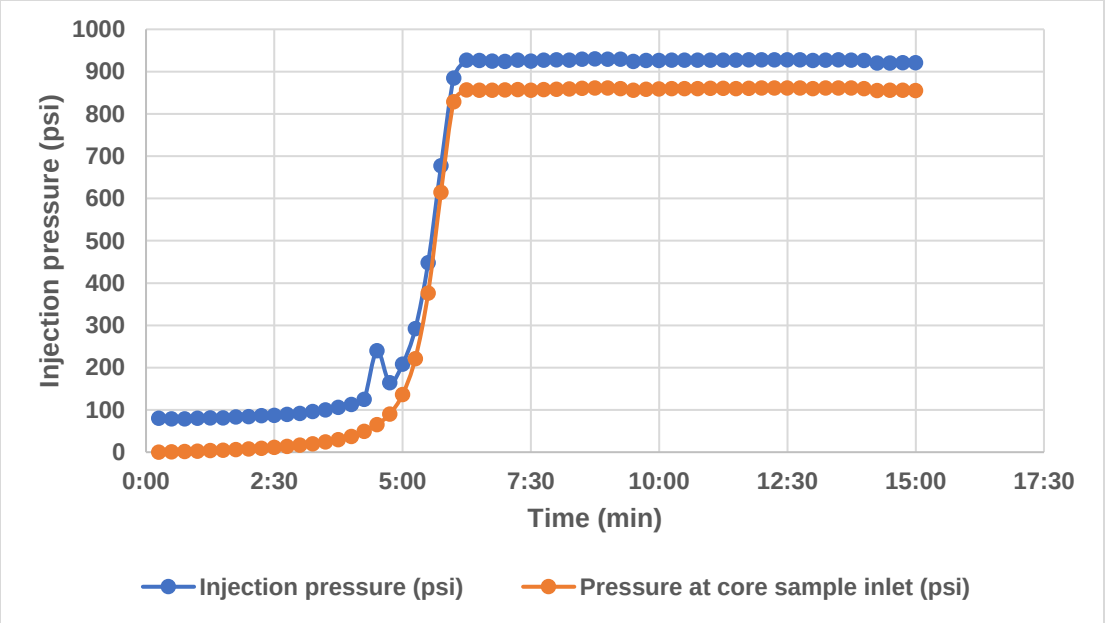

Figure 38. Pressure drop across the lines 5mL/min and 800 psi BPR

The first experiment was conducted under 800 psi back pressure and a 5 mL/min flow rate. It took 5 minutes for the pump to fill the system and build pressure to surpass the overburden pressure. Injection pressure increased from 208 to 927 in 1 minute and 15 seconds. Pressure at core holder's inlet increases in the same rate as the injection pressure. The average difference from the injection pump pressure to the pressure entering the core was 71.83 psi for the entire experiment. This can be expressed as the pressure required to move the piston as the length of the tubing from the accumulator to the core holder is relatively low having only 41" of tubing.

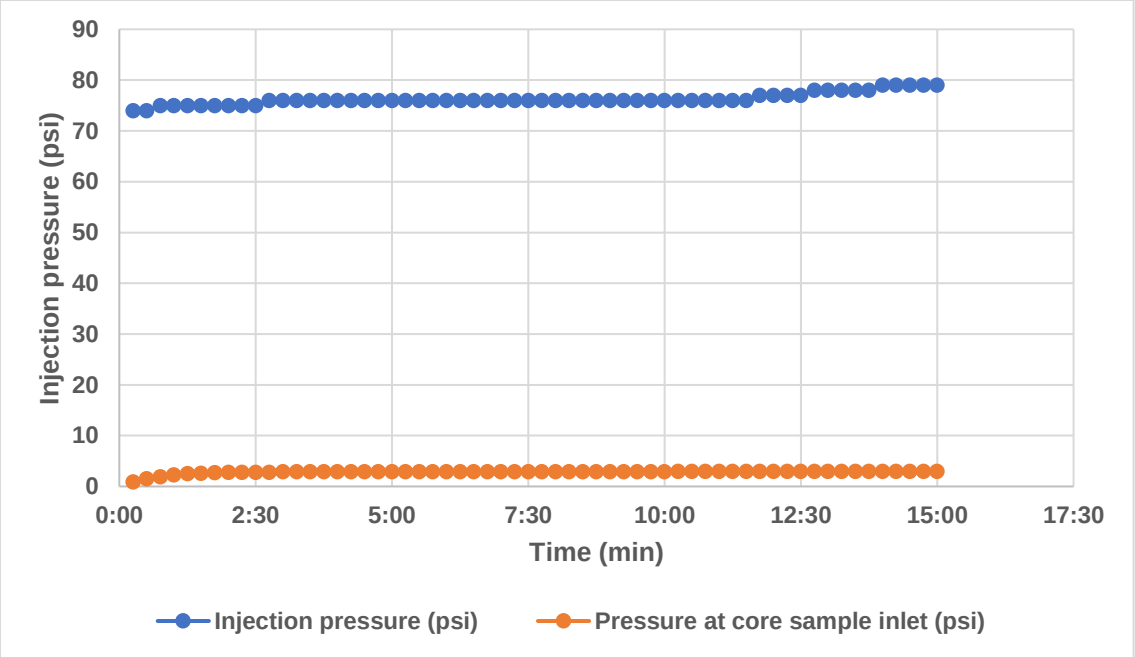

Figure 39. Pressure drop across the lines 5mL/min and 0 BPR

A second test was performed with the same flow rate but no back pressure to test if the setup, and the transducers are consistent at different back pressures and flow rates. The average difference between injection pressure and core holder inlet's pressure was 73.45 psi through the entire experiment. Compared to the average with back pressure, there is a 2-psi fluctuation that can be caused by the piston.

To compliment and confirm the results previously obtained, a 10 mL/min flow rate test was performed using both back pressure and no back pressure combinations. The pressure drop from injection to the core holders inlet was 77.4 psi average for the test with no back pressure. For the 400 psi back pressure there was a 74.56 psi average pressure drop. It is observed that pressure drop is higher at higher rates and this is caused due to the resistance of the piston at higher speeds, also, as there are 6-90° pipe curves, the higher the speed, the higher the fluid pressure loss caused by friction.

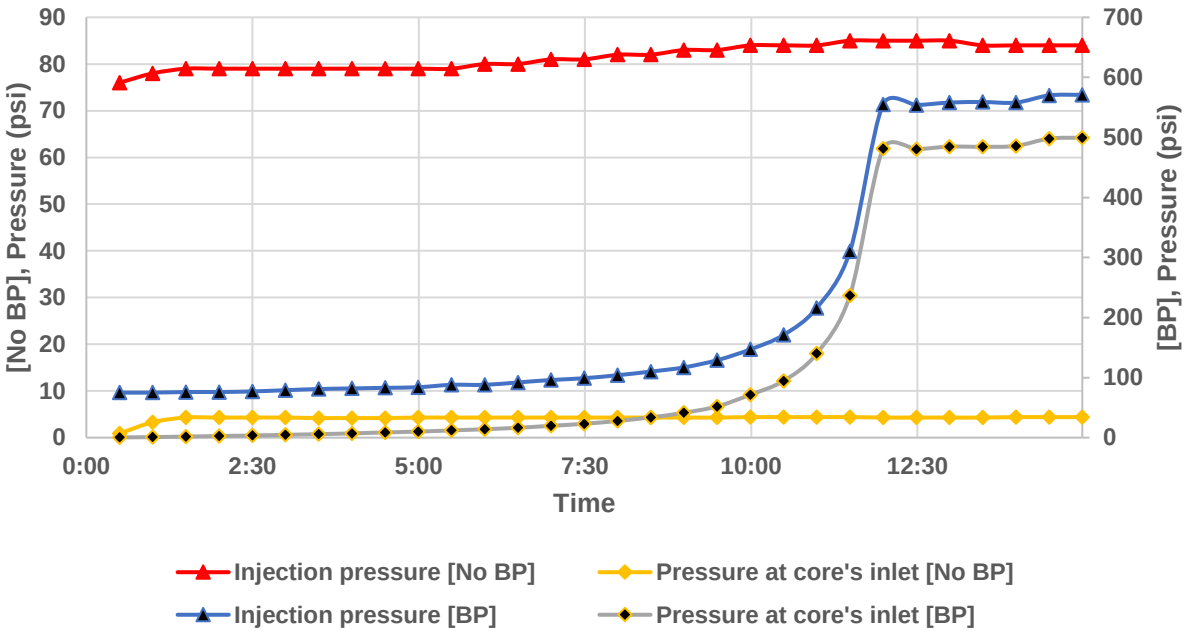

Figure 40. Pressure drop comparison

5.6 Steady state

A test for permeability and steady state was performed to evaluate the laboratory's results at different flow rates and back pressures. No acid was utilized, only water was injected as steady state does not require acid injection. One sample was tested at three different flow rates, 2, 5 and 10 mL/min. The sample's length is 1.448" and a 1" diameter. Dry weight was recorded as 41.488 grams. The 10 mL/min flow rate injection did not have back pressure and overburden was maintained 300 psi above the core holder's inlet.

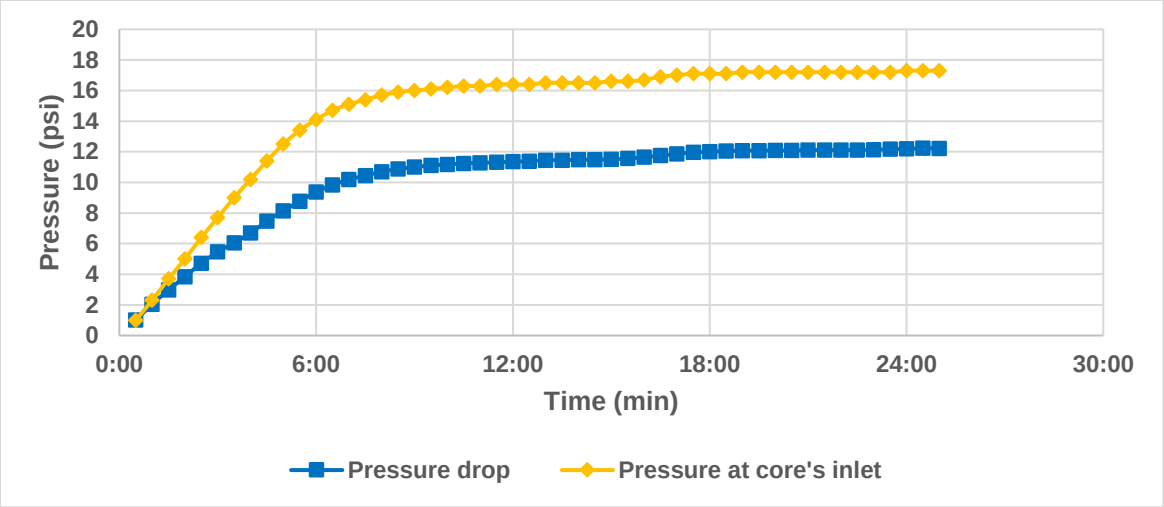

Figure 41. Steady state 10 mL/min pressure vs time

Pressure drop and core's entering pressure present the same behavior, as no back pressure is applied, it only requires a difference of 12 psi from top to bottom to allow steady fluid flow. The pressure at the core's inlet reaches a 16-17 psi range after 9.5 minutes and then stays constant for the next 15.5 minutes.

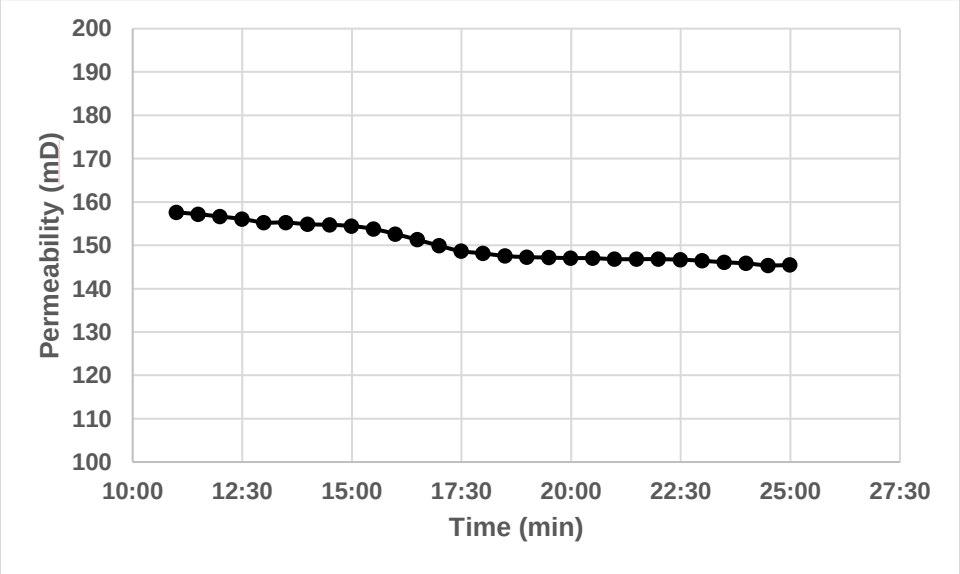

Figure 42. Permeability vs time 10 mL/min

Permeability is automatically plotted by using Darcy's lay for linear flow and with the laboratory adapted equation. After 9.5 minutes that steady state has been reached, permeability yields an average value of 146 mD, this is considered as a high permeability rock, and it is highly suitable for acidizing. Diversion occurred after 2 minutes and exit fluid flow was consistent to 10 mL/min inlet flow.

Syringe pump and accumulators were refilled, and injection flow rate was lowered to 5 mL/min. Sample stayed in the core holder and lines where drained from all the water leftovers. Back pressure was increased to 500 psi.

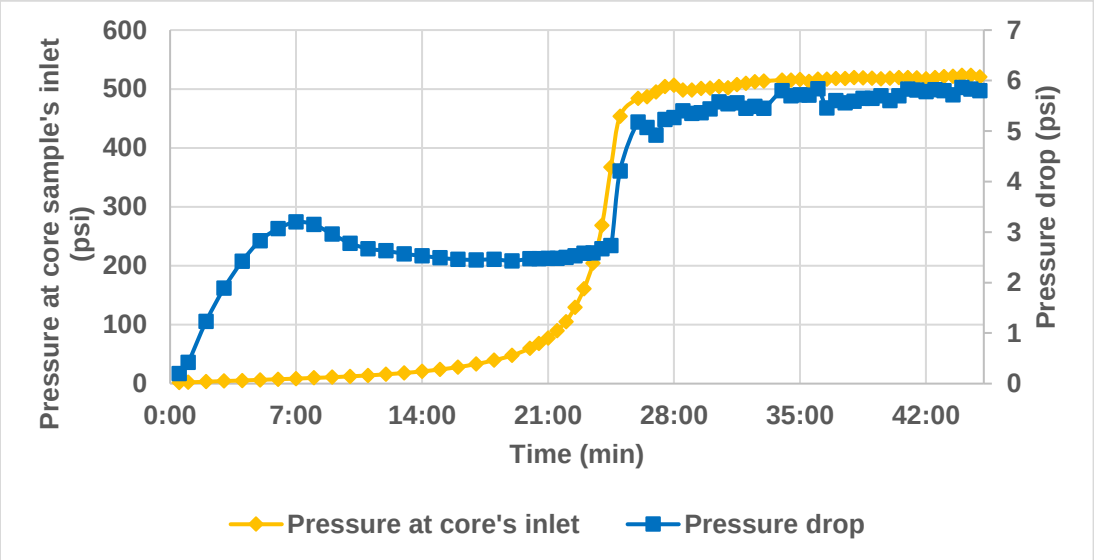

Figure 43. Steady state 5 mL/min pressure vs time

Figure 43 shows the pressure at the inlet of the core sample and pressure drop. It can be observed a drop in the pressure drop at minute 7. As the utilized sample was 1.5" long, a spacer was placed at the bottom of the sample to fill out the remaining space. This spacer requires a fluid volume of 1.82 mL to fill. Pressure starts building for the first seven minutes as the fluid requires certain pressure to flow through the rock. After 7 minutes fluid has flower through the rock but instead of immediately reaching the back pressure regulator and build pressure, it

reaches the spacer, which requires time to fill along with the bottom lines. This is described as the pressure on the bottom building and a decrease in pressure drop. It took an approximate of 23.5 minutes to start increasing the injection pressure from 280 to 541 psi after 25 minutes. Diversion occurred at 27 minutes when pressure at the core holder's inlet was 506.5 psi. Volume obtained at the exit port was constant and matched the injection volume displayed by the syringe pump. Pressure drop follows the same pattern and stabilizes at 5.7-5.8 psi. Calculated permeability was 152 mD after the water reached steady state.

The last test performed had a flow rate of 2 mL/min and 600 psi back pressure. Overburden was maintained approximately 300 psi above injection pressure. Total experiment duration was 132 minutes

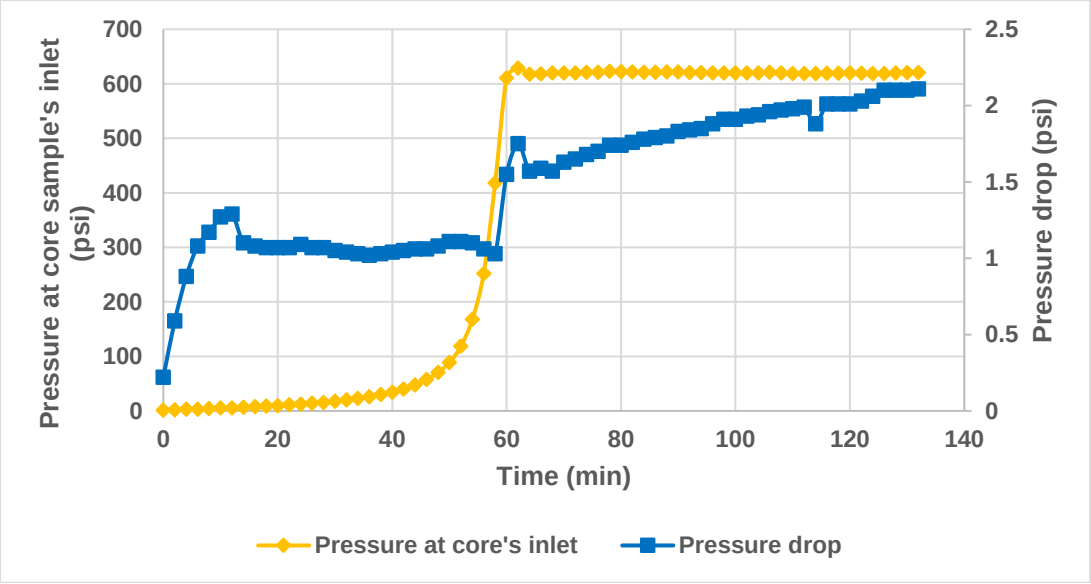

Figure 44. Steady state 2 mL/min pressure vs time

Pressure at the core sample's inlet presented a slow increase for the first 30 minutes and slowly increased from 17.8 to 610.6 psi after 60 minutes of test. It remained constant for an hour with an input pressure of 619-620 psi. Diversion was reported after 60 minutes once the back

pressure was surpassed by 10.6 psi. The volume of the fluid exiting the core was measured multiple times for random periods of time. The observed volume matched the inlet volume reported by the syringe pump. Pressure drop was only 1.8-2.1 psi from minute 80 to the end of the test. Calculated permeability is 169 mD.

CHAPTER VI

SUMMARY AND CONCLUSION

A functional core flooding laboratory was designed, assembled, configured, and tested. The laboratory has proven to be suitable for matrix acidizing purposes. The design entire system was designed to support pressures up to 5,000 psi, however most of the components can reach higher pressures. The main setup limiting factors are the differential pressure transducers as the absolute pressure rating is 3626 psi. Surpassing this pressure will cause irreparable damage to the transducers. However, two-way valves have been installed on all transducer ports to close the flow and prevent fluid from entering in case the system is intended to surpass this pressure.

The laboratory can run under a high flow rate range from 0.001 to 107 mL/min. It can also support testing at temperatures as high as 250 ˚F. Tubing has been optimized to a 36 ft setup including all connections and components. A hardware/software connection was performed, and communication was established between the pressure transducers and LabVIEW software. Laboratory was built to be ergonomically used by any person. Frame movement is achieved as it can easily be moved to any place without any constraint other than the nitrogen gas connection. Furthermore, a detailed description of all the components, technical specifications and material properties were included for future modifications and testing compatibility with different materials. A full and detailed description of the laboratory's usage was also developed to help the users to be able to run the laboratory from the beginning to the end without previous experience

and get the desired consistent results. A set of procedures, equations and data analysis help to understand the rock properties and behaviors. Pressure transducers were also calibrated and designed to receive information from different pressure points.

To ensure laboratory's functionality, repeatability and conciseness, setup was modified multiple times to test different sections and equipment performance such as back pressure, overburden pressure, injection pressure, steady state and pressure drop across the lines. From the steady state tests at 10, 5 and 2 mL/min flow rates, it can be realized that calculated permeability increases as the flow rate decreases increasing from 146 mD at 10 mL/min to 169 mD at 2 mL/min. This discrepancy is mainly caused because of the sample's size being 1.4" in length. The suggested lengths for a better data acquisition are from 4-6" as the fluid will have more rock interaction and values will be more accurate. It was also proved that there is a difference between injection pressure and actual pressure entering the core caused by the friction of the piston in the accumulators. Acid was successfully injected to low permeability limestone and to a non-carbonate rock and the results were analyzed. Finally, the importance of a right overburden pressure application and its requirements were covered by performing multiple tests at different overburden pressures. The overall laboratory was proven to be functional and is ready for future testing.

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APPENDIX

APPENDIX

Table 6. Core flooding valve configurations

Injection pump refilling	
Valve #	Configuration
5	A

Injection pump release pressure	
Valve #	Configuration
6	Open

Acid refilling	
Valve #	Configuration
1	Closed
2	B
3	B
5	A
6	Open
7	Open

Brine Injection	
Valve #	Configuration
4	B
5	B
6	Closed
7	Closed
8	Open
9	B
10	A

Brine refilling	
Valve #	Configuration
1	Closed
2	A
4	A
5	A
6	Open
8	Open

Acid injection	
Valve #	Configuration
3	A
5	B
6	Closed
7	Open
8	Closed
9	B
10	A

BIOGRAPHICAL SKETCH

Antonio Hernandez Zuniga was born in Reynosa, Mexico, in March of 1996. He studied in Mexico until High School in 2014 where he got awarded as valedictorian. He started his college studies in June 2015 at University of Texas Rio Grande Valley in Edinburg. He pursued his bachelor's degree in Manufacturing Engineering. He performed multiple roles such as Chemistry tutor, Calculus tutor, Engineering Economics, etc. He was also involved in many organizations such as SME and SHPE. He graduated in May of 2019 and decided to pursue his Master of Science in Mechanical Engineering at University of Texas Rio Grande Valley working in a development of a matrix acidizing core flooding laboratory. After two and a half years, he earned his Master of Science in Mechanical Engineering in December 2021. If any question arises, please contact him to 956-340-9578 or Antonio_zts1@hotmail.com.