Investigation of Permeability Deformation of Class G Cement During Acidizing Treatment

by

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14902

Dissertation submitted in partial fulfilment of
the requirement for
Bachelor of Engineering (Hons)
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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Petroleum Engineering Programme
Universiti Teknologi PETRONAS
In partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(PETROLEUM)

Approved by,

____
(Dr. Syahrir Ridha)

UNIVERISITI TEKNOLOGI PETRONAS
TRONOH, PERAK
January 2015

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done unspecified sources or persons.

MUHAMMAD SHAMIR BIN ABDUL RAHIM

ABSTRACT

Class G cement is widely used in oil and gas industry for well cementing. When the well cannot produce the hydrocarbon as per demand due to positive skin, the well stimulation is carried out. One of the popular well stimulation is acidizing treatment. Previous studies have shown that acidizing treatment was affected the cementing of the wellbore which led to the zonal isolation problem. Even though the main purpose of applying the acidizing treatment is to enhance or improve the productivity of producing hydrocarbon unfortunately it also gives the bad impact to the oil and gas industry as well. The objective of this study is to investigate the permeability deformation of Class G cement during acidizing treatment. Throughout the project, a comparison study between before and after the analysis was carried out. The aims of analysis are to find out the mass loss and also to investigate the permeability deformation upon acid stimulation. The first step was to cure the cement slurries under condition of 3000 Psia and 90°C based on average well condition in Malay basin for eight (8) hours. Then, the samples were immersed in three (3) different concentrations of hydrochloric acid (HCL) / hydrofluoric (HF) acid solution or known as mud acid for 40 minutes. The samples were analysed both before and after immersed into acid solution. The analysis included mass loss due to acid stimulation and permeability deformation by using Permeameter and Porosity Meter (POROPERM). Result from POROPERM showed that the permeability of cement sample increased after the acid stimulation where the higher concentration of acid, the higher the permeability. From the result gathered, it can be concluded that the acid used in acidizing treatment affected the permeability of Class G cement.

ACKNOWLEDGEMENT

First and foremost, I would like to thank Allah S.W.T. for giving me the opportunity to complete my final year project entitled Investigation of permeability deformation of Class G cement during acidizing treatment.

This project would not successfully complete without the help of many people and here I would like to express my sincere appreciation to Dr. Syahrir Ridha, Lecturer of Petroleum and Geosciences Faculty for his valuable guidance and advice as my final year project supervisor. My special recognition I bid to Technologist in Petroleum Engineering Laboratory, Mr. Khairul Nizam and Mr. Jukhairi for their help and guidance in handling Class G Cementing experiment. Not to forget, to Mrs. Hilmayeni for her help in assisting me using the Permeameter and Porosity Meter (POROPERM) and Mr. Zulhusni for his guidance and advice in how to deal with acid in order to prepare acid solution.

Special thanks to my family members for their supports as well as everyone whose helping and sharing me their valuable knowledge.

Thank you.

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LIST OF ABBREVIATIONS

API	American Petroleum Institute
Al	Aluminium
Ca	Calcium
$CaCl_2$	Calcium Chloride
CaF_2	Calcium Fluorite
Ca(OH)	Calcium Hydroxide / Portlandite
CaS	Calcium Sulfide
Fe	Iron
H^+	Hydrogen Ion
HAc	Acetic Acid
HCL	Hydrochloric acid
HF	Hydrofluoric acid
HPHT	High Pressure High Temperature
K	Potassium
Mg	Magnesium
POROPERM	Permeameter and Porosity Meter
Si	Silicon

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Cement is a binder, a substance that sets and hardens and can bind other materials together [1]. It has been used in construction industry as well as oil and gas industry for many years but both have different in the properties of cement. In an oil well, cement is being used for cementing the casing well completion. In general, there are two (2) type of well completion [2].

- Open hole completion in which no casing or liner run in the wellbore.
- Cased hole completion in which after drilling assembly were tripped out of hole, steel casing or liner will be run, cemented and perforated to create the flow of producing hydrocarbon of reservoir into the well.

Cementing is one of the most important processes in completing a well. It is a process whereby the slurry of cement, cement additives and water are mixing at the surface and pumping it down by hydraulic displacement to the desired location [3]. Then, the slurry is let to hardened, or reacted, to become a rigid solid that exhibits favourable strength characteristics. It served five (5) primary functions [4].

- Provide zonal isolation.
- Provide casing support and protection against corrosive fluids.
- Support axial load of casing string.
- Plug a portion of a well for side-tracking.
- Plug non-producing well for abandonment.

Meanwhile, well stimulation is a method applied to enhance the productivity of natural resources of the existing well by enlarge old tunnels or to create new path in the producing formation [5]. It is being applied when the formation cannot produce as per demand due to formation damage in which has altered the flow of fluids into the wellbore. Nowadays, there are many techniques that can be used to stimulate the well such as explosive fracturing, hydraulic fracturing, acidizing and steam injection [5]. It is claimed that the acidizing is the most feasible and widely used of well stimulation techniques since it can performed in both sandstone and carbonate formation [6]. Moreover, compare to the other available well stimulation techniques, acidizing can be classified as one of the most simplest and economical technique to enhance the productivity [7].

Acidizing treatments are divided into two categories: matrix acidizing and fracture acidizing. It can increase the production in many well if properly applied by having an understanding of the purpose of treatments and what is necessary to stimulate the well [8]. For fracture acidizing, acid is pumped into the wellbore out into the formation at pressure high enough to exceed the rock formation fracture pressure [9-11] while matrix acidizing, the acid is pumped into the wellbore out into the formation at a lower rate aiming to remove the formation damage [9,11,12]. Only matrix acidizing can be applied in both sandstone and carbonate formation but not for fracture acidizing in which can be applied just for carbonate formation mainly due to the difference in reaction mechanism between acid and formation rock [9].

Even though the acidizing treatment gives a good impact in enhance the productivity of natural resources, improper usage of the acid may pose problems to the cements and it may lead to the zonal isolation problems [5]. Hence, this study is conducted to focus at the effect of acid attack during acidizing treatment especially on the permeability changes.

1.2 PROBLEM STATEMENT

Oil well cement is affected by the acids used in acidizing treatment where it causes the zonal isolation problem [5] due to occurrences of the reactions between the hardened cement slurry in the annulus and acid from the treatment [13]. Generally, combination of hydrochloric (HCL) and hydrofluoric (HF) acid solution with additives are used in acidizing treatment to stimulate the sandstone formation [8].

A literature survey indicated that significant numbers of cement squeeze jobs were found to break down or develop zonal isolation problems after HF/HCL acidizing. For example, past experience in Prudhoe Bay field operating area shows that more than 37% of primary cementing jobs developed zonal isolation problems after acidizing and 73% of squeeze cement jobs break down after the HF/HCL acid stimulation [14]. However, there are not many researchers have studied about the effect of acid in acidizing treatment on the permeability deformation of Class G cement.

1.3 OBJECTIVE AND SCOPE OF STUDY

The primary objective of this project is:

- To investigate the permeability deformation of Class G cement during acidizing treatment in reservoir condition.

The scope of study that will be focused in this project:

- Study the effect of acid solution on physical condition and mass loss of Class G cement.
- Study the concept of acidizing treatment and influence of difference concentration of acid solutions in permeability deformation of Class G cement.

CHAPTER 2

LITERATURE REVIEW

Based on the literature survey, acidizing treatment affected the oil well cement even though the main purpose is to increase the production of producing hydrocarbon. A significant number of studies about the cement resistance to the attack of acid during acidizing treatment have been done and the results proved that the acids were affecting the cement physically and chemically.

Many factors have been found in the previous study that contribute to the cement resistance to the acid attack such as the chemical composition of the cement, cement slurry composition, acid solution and curing condition referring to the temperature and pressure in the preparation of cement [5, 13].

In order to identify the chemical composition of the cement, complexometric and thermogravimetric analyses were performed. This is to determine the carbonate content and pre-hydration of the cement samples. From the result of analyses, it was found that the carbonate content in the cements is consistent with the observed difference in the mass loss of cement by acids. Generally, cement contains several types of carbonate elements. A greater level of Calcium Sulfide (CaS) consequently yields a higher level of portlandite $Ca(OH)_2$ which being soluble in HCL and results in a large degree of acid attack toward cement [13].

In preparing the cement, the specific amount of cement and water are required and it must be mixing together using speed mixer by following the step in API recommended practise 10B-2 [15] then it will be cured. Curing is a process in which the cement is protected from loss of moisture and kept within a reasonable temperature range. It is found that in the study, when the cement is cured under high temperature and pressure, the degree of hydration will increase which result in smaller and more evenly distribution of $Ca(OH)_2$ crystals. This provide effective barrier to the acid attack because the cement strength is increase while the permeability is decrease [5].

FIGURE 2.1 indicated that the mass loss decrease as the curing condition increase. It is due to the chemical components are more distributed and uniform at high temperature. This will result the decreasing cement permeability that control the rate of acid attack toward the cement.

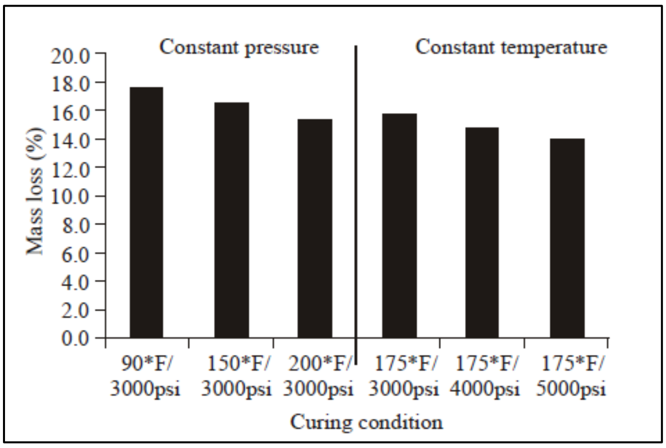


FIGURE 2.1 Mass loss (%) with different curing condition [5]

FIGURE 2.2 showed that the depth of penetration of acid in sample 1 is the highest which is 158.2 μm while the sample 6 had the smallest depth of penetration with 25.86 μm in length. It shows that, when curing condition is increase, the chemical components are more distributed result the decreasing the cement permeability which provide a good barrier to acid attack.

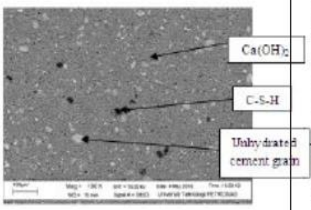
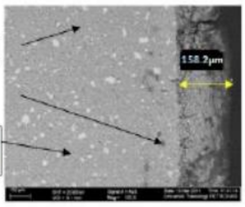
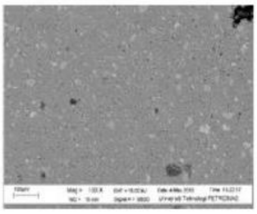
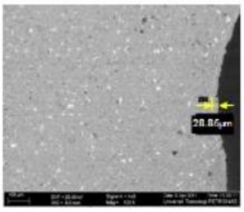
Sample	Before	After
Sample 1 (cure under 90°F and 3000psi)		
Sample 6 (cure under 175°F and 5000psi)		

FIGURE 2.2 BEM-SEM image of Class G cement cured under different temperature and pressure condition [5]

A new approach has been used to reduce the level of acid attack on the hardened cement which is adding latex cement additive into cement slurry. It has been used successfully across a wide range of well condition and is used in both primary casing cementing and remedial squeeze work. An experiment has been conducted and the result shows that the mass loss caused by the acid with latex is less than the cement without latex [13].

Acid is a corrosive substance where it will destroy and damage other substances with which it comes into contact. In acidizing treatment, acid is being used to stimulate the producing formation [8] and it comes into contact with the cement casing. In a long term, the cement casing could be damage due to acid attack. A few tests of varies type of acid mixture has been conducted to observe the degree of acid attack on hardened cement. In this study, the following mixture has been used; 10% HAc, 10% HAc / 1.5% HF and 12% HCL / 3% HF. Portland Class G cement is being used in this test and the cement solubility was observed. 12% HCL / 3% HF mixture was shown to be the most aggressive to the cement, follow with 10% HAc and, 10% HAc / 1.5% HF.

TABLE 2.1 Influence of the acid solution on the resistance of the cement cubes - mass loss and composition of the acid solution [13]

Test	ACID	MASS LOSS %	Ca	Si	Fe	Mg	K	Al	S
1	12% HCL / 3% HF	12	518	408	139	70	38	0	38
2	10% HAc	2	698.5	10	24	12	25	11	0
3	10% HAc	2	1000	14.6	37	20	25	15	11
4	10% HAc / 1.5% HF	0	36	19	3	0	16	0	0
5	10% HAc / 1.5% HF	0	0	32.1	1.6	0	21	0	4.6
Conventional cement slurry Curing conditions: T: 52°C, P: 20.7 MPa									

Table 2.1 shows that, the 12% HCL / 3% HF results the highest mass loss of cement which is 12%, follow with 10% HAc is 2% and 10% HAc / 1.5% HF approximately to 0%.

The difference mass loss of cement by acid solution is attributed to difference in solubility of fluorite where it is much more soluble in HCL compare to HAc. The superficial layer of fluorite tends to remain adhered to the surface of the cement in cases where the attack is by mixtures of HAc/HF, which in turn tends to prevent further attack by the acid. In the case of HCL/HF mixtures, fluorite layer is readily solubilized in the solution, expose cement surface which is attacked, leading to a greater mass loss. The different type of acid used also affecting the cement.

CHAPTER 3

METHODOLOGY

3.1 RESEARCH METHODOLOGY

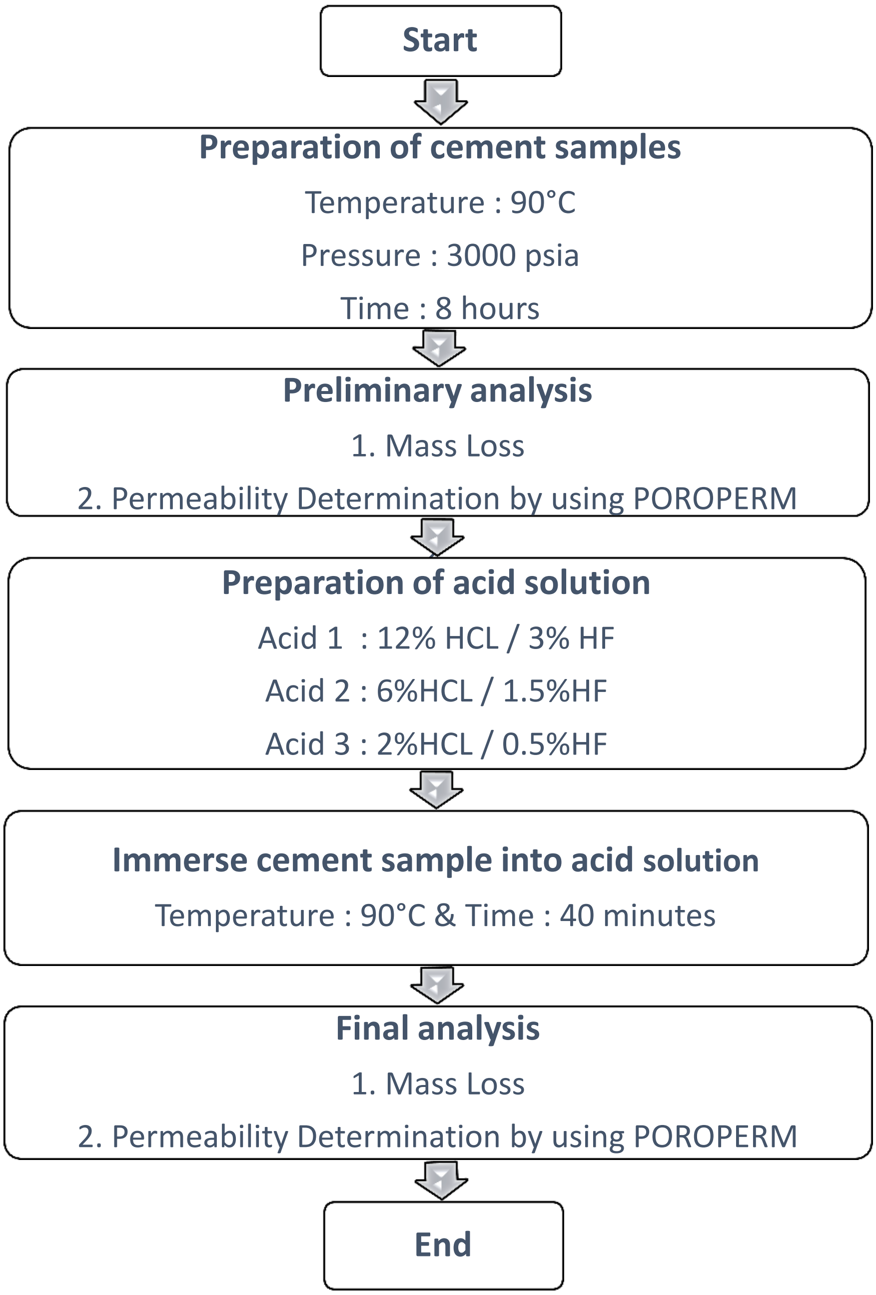


FIGURE 3.1 Experiment procedures

3.1.1 Preparation of cement samples

The first step in the experiment procedure was to prepare Class G cement samples. It was done by mixing the calculated amount of cement and water using the constant speed mixer. The ratio of water to cement used in the cement samples preparation was 44% according to API Recommended Practice 10B-2. 200g of Class G cement and 88ml of distilled water approximately were needed to prepare for one cement sample.

FIGURE 3.2 Constant Speed Mixer

Then, the cement slurries were poured into the cubic mold and cured for eight (8) hours at pressure of 3000 psia and temperature of 90°C in the High Pressure High Temperature (HPHT) curing chamber. The curing pressure and temperature were selected based on the average temperature and depth of Malaysia oilfield. The (8) hours of curing duration was selected because in most cases, it was the minimum time for the cement to develop the minimum compressive strength in a well which is 500 psia [2].

FIGURE 3.3 High Pressure High Temperature (HPHT) curing chamber

After curing process was done, the samples were examined. Only the best samples were taken for preliminary analysis. The purpose was to minimize the error in conducting the experiment.

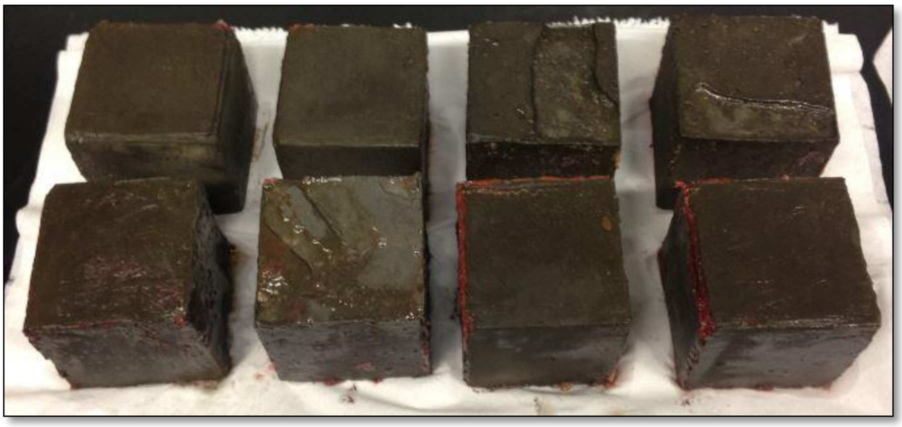


FIGURE 3.4 Class G cement samples after curing

3.1.2 Preliminary analysis

In this study, two (2) preliminary analysis were conducted which are mass loss and permeability determination by using Permeameter and Porosity meter (POROPERM).

Mass Loss

The cement samples were weighed before and after being immersed into acid solution. It was used to calculate the mass loss due to acid solution. This is necessary to quantify the consumption of the cement by acid with different concentration.

Permeability Determination

Permeability determination analysis was conducted to determine the permeability of cement samples by using the POROPERM instrument. This instrument was injecting Helium gas pass through the sample to check the permeability of it. The sample was been weighed, been measured the diameter and the length. These data was been input in the software before running the test.

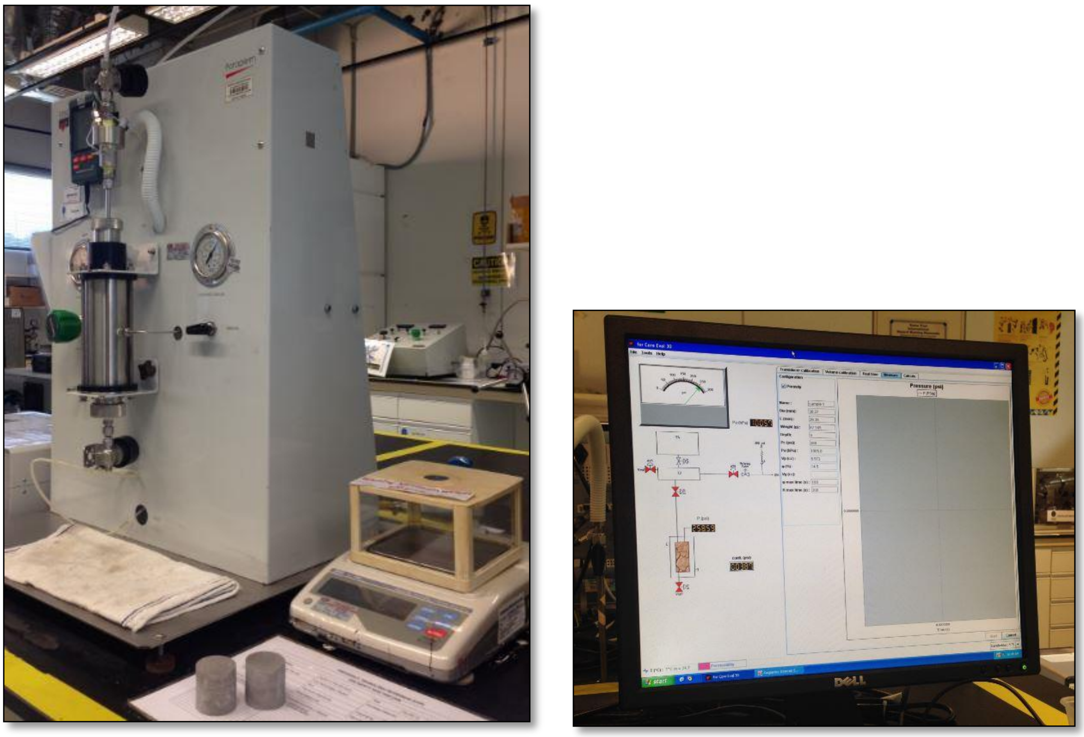


FIGURE 3.5 POROPERM, Weighing Scale & Data display

Prior to permeability testing, the cured cement samples were cored in order to have a size of POROPERM holder. It was cored using core cutting saw to be in cylindrical shape. Before coring, the cured cement samples were surrounded by other cement which acts as a holder.

Procedures of Core Cutting

i. The cement sample was placed in the core cutting saw holder.
ii. The switch is on.
iii. The water supply was opened which act as lubricant during the coring process.
iv. The rotating cutter was pulled down slowly to cut the cement sample and get a cylinder shape for cured cement.

Then, the core cement samples were trimmed with core trimming saw machine to have a better shape and at least 1.0 inches in length.

Procedures for Core Trimming

i. Cement sample was placed in the core trimming holder.
ii. The switch is on and water supply was automatically on which act as lubricant during trimming operation.
iii. The trimming process was started by pushing the cement holder through the saw.

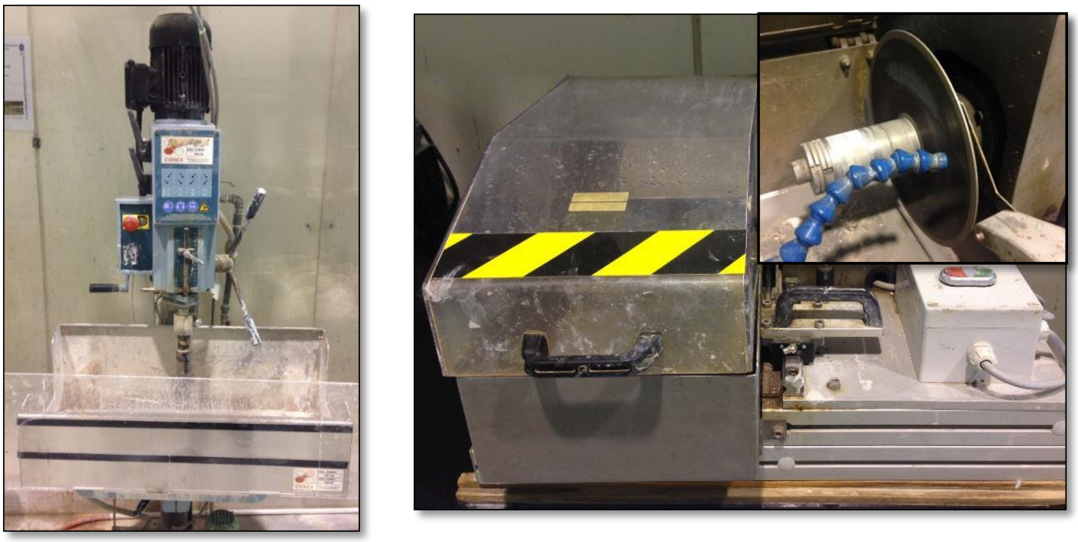


FIGURE 3.6 Coring Machine & Trimming Machine

FIGURE 3.7 Core cement samples

3.1.3 Preparation of acid solution

The objective of acid solution preparation was to stimulate the downhole condition during acid stimulation in a well. In this study, the acid was prepared by mixing calculated amount of hydrochloric acid (HCL) and hydrofluoric acid (HF). Three (3) different concentration of acid were prepared for testing. This combination of acid was known as mud acid. The ratio of concentration of HCl to HF is 4 to 1. Mud acid is chosen because this combination of acid solution is widely used in this industry of acidizing stimulation especially in sandstone formation. The concentrations of acids used are listed in **TABLE 3.1** below;

TABLE 3.1 Acid concentrations

Acid	Acid Concentration
1	12 % HCL / 3 % HF
2	6 % HCL / 1.5 % HF
3	2 % HCL / 0.5 % HF

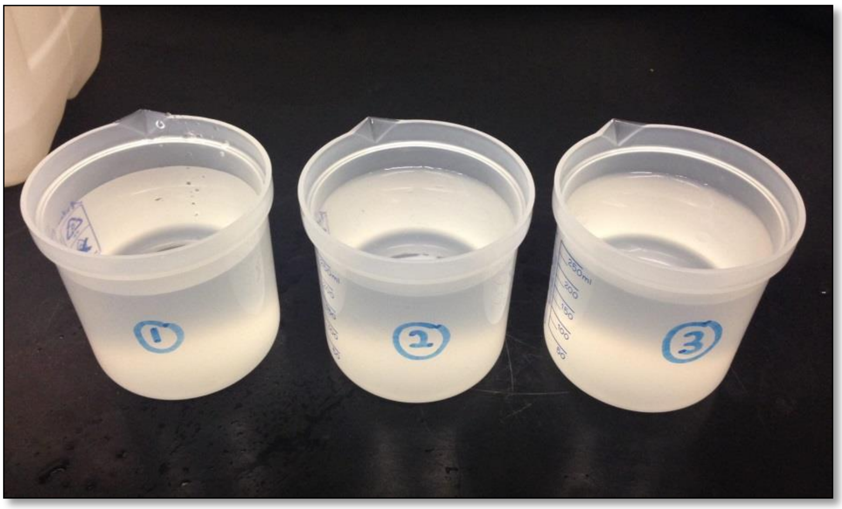


FIGURE 3.8 Acid solutions with different concentrations

3.1.4 Immerse of cement samples into acid solution

The cement samples were immersed into the acid solutions at the temperature of 90°C for 40 minutes. In order to meet the desired temperature for acid solution, the thermostatic bath was used to heat the acid solution. A beaker contain of 0.25L of acid solution was placed in thermostatically controlled water bath. Since the acid solution involved with hydrofluoric acid (HF), a polypropylene beaker was used rather than a glass beaker because hydrofluoric acid (HF) can have a reaction with glass beaker and resulted in less reaction with the cement sample later.

Due to the sensitivity of the thermocouple, the reading on the monitor might fluctuate. Thus, to ensure correct temperature of the solution inside the beaker, a thermometer needed to be placed inside the beaker to constantly check the temperature of the solution. However, due to the fact that hydrofluoric acid can have a reaction with glass, the second beaker with the same volume of water placed alongside the first beaker as the temperature controller. It was done because HF can have a reaction with the glass of the thermometer. Then, the acid solution was heated and reached desired temperature which is 90°C. The cement sample immediately placed in the beaker of acid and left about 40 minutes for exposing purpose. The exposure period of 40 minutes was selected based on the average acid spending time at the formation before it pumped back to surface.

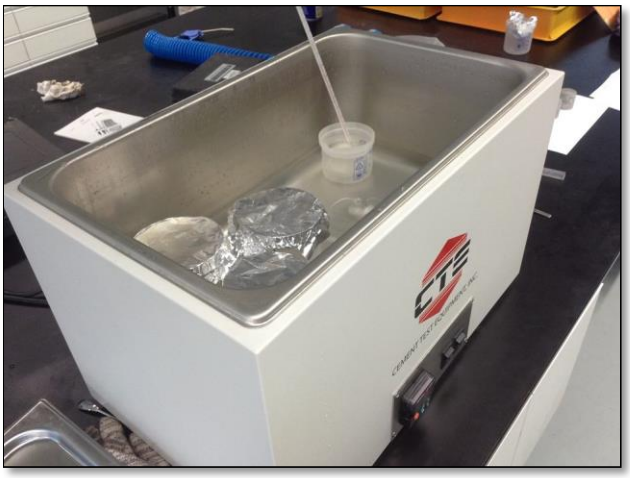


FIGURE 3.9 Thermostatic Bath

3.1.5 Final analysis

Lastly, after 40 minutes the cement samples were left immersed in the acid solution with temperature of 90°C, the cement samples were removed and allowed to dry at room temperature for approximately one (1) to two (2) hours. The cement samples were then weighed and the final mass was recorded. The cement samples were left to dry again for another hour and been weighed again. The purpose of this repeating weighing process was to see if the cement samples are still reactive to the residual acids in the cements or not. The mass loss for the cement samples due to effect of acid solution was calculated by the difference between initial mass and the final mass of the cement samples.

$$\text{Mass loss (\%)} = \frac{(\text{ Initial Mass} - \text{Final Mass})}{\text{Initial Mass}} \times 100$$

The immersed cement samples undergo the same testing as in the preliminary analysis. The purpose of final analysis was to compare the condition of samples with its initial condition before being immersed into the acid solution. The analysis that was conducted is Permeability Determination by using POROPERM.

3.2 KEY MILESTONE

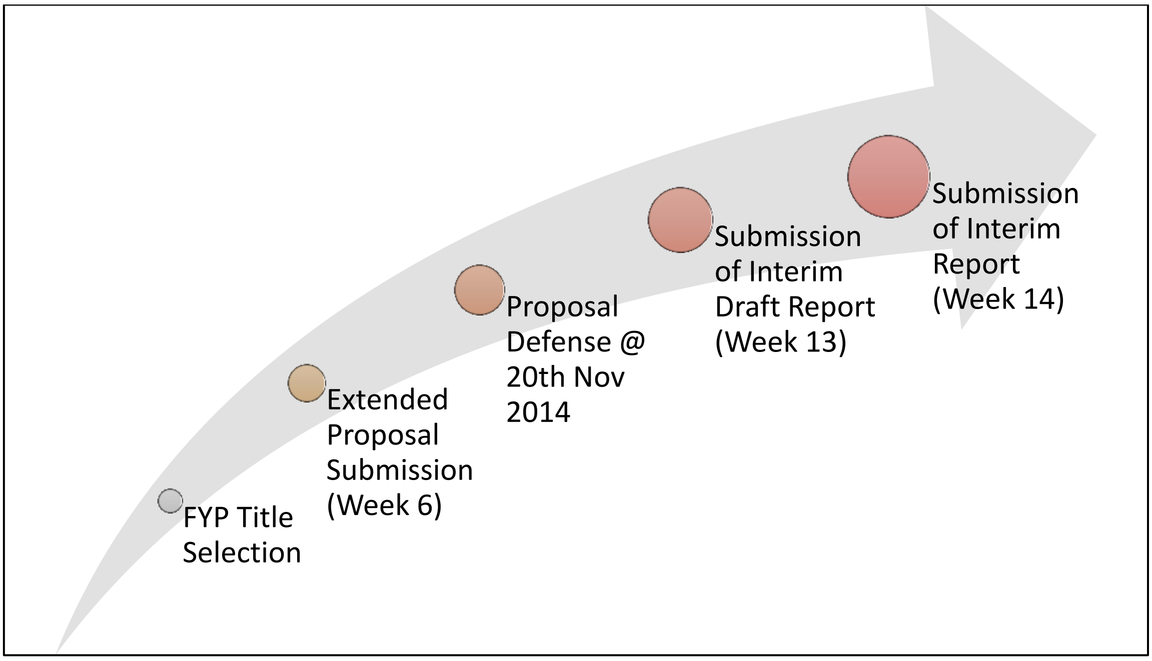


FIGURE 3.10 Key milestone of FYP 1

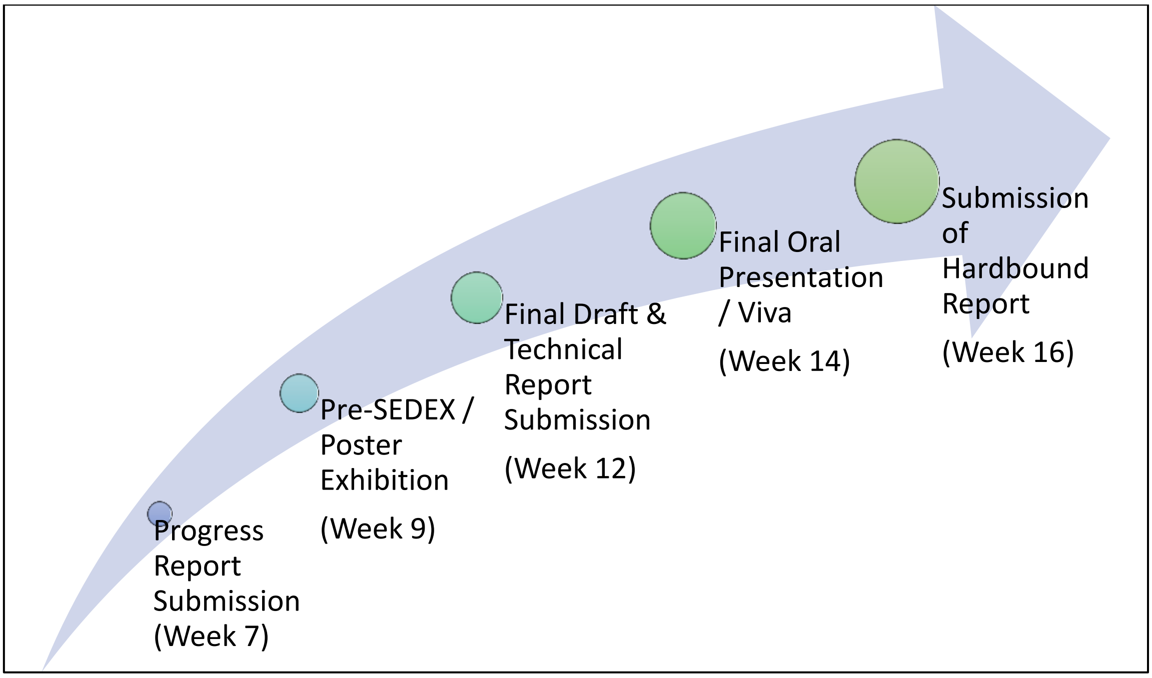


FIGURE 3.11 Key milestone of FYP 2

3.3 TOOLS, APPARATUS & MATERIALS REQUIRED

TABLE 3.2 List of Tools/Apparatus and Materials used to complete the project

Tool/Apparatus	Function
Constant Speed Mixer	To mix the Class G cement and water
High Pressure High Temperature (HPHT) Curing Chamber	To cure cement slurries
Core cutting saw & Core trimming saw machine	To cut and trim the cement sample in order to have a cylindrical shape
Permeameter and Porosity Meter (POROPERM)	To determine the permeability of cement samples
Thermostatic bath	To heat the acid solution
Polypropylene Beaker	To keep acid solution
Materials	
1. Class G cement 2. Acids (Hydrochloric Acid Solution, Hydrofluoric Acid solution)	

3.4 GANTT CHART

TABLE 3.3 Gantt chart of the planned schedule of the project

Activities of the week	FYP 1														FYP 2													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Selection of Project Title	▨	▨																										
Material or Journal Findings / Data Gathering		▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨																
Proposal Defense									▨																			
Submission of Draft Interim Report													▨															
Submission of Interim Report														▨														
Cement Sample Preparation															▨	▨	▨											
Preparation of Acid Solution																				▨	▨							
Immersed Cement into Acid Solution																						▨	▨					
Permeability Determination Analysis																					▨	▨	▨	▨				
Result and Final Findings																								▨	▨			
Final Report Submission and Viva																										■	■	■

Done	▨
In Progress	■

CHAPTER 4

RESULTS AND DISCUSSIONS

In this chapter, the results were discussed based on the objective stated in the introduction part which was to investigate the permeability deformation of Class G cement during acidizing treatment. It was divided into three (3) parts; physical appearances, mass loss and permeability of Class G cement before and after immersed into acid solution.

4.1 PHYSICAL APPEARANCES

BEFORE	AFTER
	

FIGURE 4.1 Class G cement physical condition before (left) and after (right) immersed into acid solution.

Based on the **FIGURE 4.1,** the grey cement cubes on the left showed the initial physical condition of Class G cement sample before being immersed into acid solution while the cement sample on the right showed the physical condition after being immersed into acid solution. The other two more samples of Class G cement that have been treated with different concentration of the mud acid exhibit almost similar physical condition. The white milky substance on the surface of sample was Calcium Fluorite (CaF_2) and Calcium Chloride ($CaCl_2$) and it was expected outcome from the reaction of acid solution with the calcium of the cement.

4.2 MASS LOSS

The mass loss of the cement in this project represented the solubility of the cement in the acid solution. The initial mass of cement samples were taken and recorded. After the cement samples were immersed into the respective concentration of acid solutions at elevated temperature of 90°C for 40 minutes, it was left to dry and been placed into oven with temperature of 90°C before being weigh. This is to make sure zero residual acid in the cement samples.

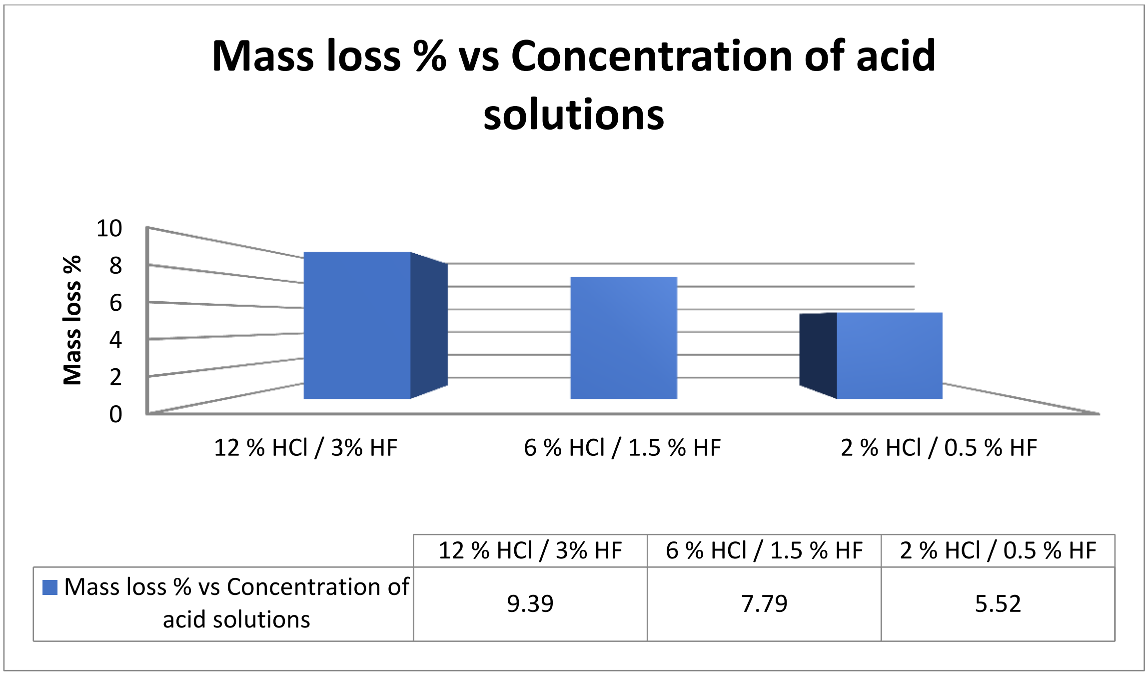
TABLE 4.1 and **FIGURE 4.2** showed the results of mass loss for each cement samples after being immersed into respective acid solutions. It can be seen that the solubility or mass loss of cement samples decreased with the decreased in acid concentration. This is due to the fact that the amount of hydrogen ions (H^+) in the acid solution decreased as the concentration of acid decreased.

Strength of acid is depending on the type of acid. In this study, combination of hydrochloric acid and hydrofluoric acid were used but with difference concentration. As the concentration of the acid increases, there are more H^+ per unit volume in the solution that can be donated or reacted which results in greater reaction between acid and cement. This explains why the mass loss increases as the concentration increases.

TABLE 4.1 Mass loss for different concentration of acid solutions

Sample	Concentration of acid solutions	Weight (g) Before	Weight (g) After	Mass Loss (%)
1	12 % HCl / 3% HF	72.542	66.314	9.39
2	6 % HCl / 1.5 % HF	70.309	65.228	7.79
3	2 % HCl / 0.5 % HF	69.808	66.159	5.52

$$\text{Mass loss (\%)} = \frac{(\text{ Initial Mass} - \text{Final Mass})}{\text{Initial Mass}} \times 100$$



	12 % HCl / 3% HF	6 % HCl / 1.5 % HF	2 % HCl / 0.5 % HF
■ Mass loss % vs Concentration of acid solutions	9.39	7.79	5.52

FIGURE 4.2 Bar chart of mass loss % vs concentration of acid solution

4.3 PERMEABILITY DEFORMATION

Study of the permeability deformation of Class G cement samples were carried out to prove that the acid used in acidizing treatment affected the permeability of cement. In this study, different acid concentrations were prepared to measure the degree of permeability deformed. The Class G cement samples were analysed using Permeameter and Porosity Meter (POROPERM).

Before the tests were carried out, all the cement samples were placed into oven for a few hours with temperature of 90°C. This is to make sure the samples were completely dried so that it can be measured by POROPERM to check the permeability. The samples were lighter after been placed in the oven compare to before been placed in the oven.

FIGURE 4.3 Roller Oven

TABLE 4.2 Permeability of Class G cement (md) before and after Immersed into acid solution

Sample	Permeability of Class G cement (md)		Increment (md)
	Before	After	
1	0.122	2.559	2.437
2	0.087	0.821	0.734
3	0.071	0.279	0.208

Based on TABLE 4.2, it showed that the permeability of Class G cements for all samples increased after being immersed into acid solutions. Sample 1 showed highest increment in permeability which is 2.437md, followed with sample 2 showed permeability increment of 0.734md and lastly sample 3 with 0.208md. From these result, it showed that when the concentration of acid solution increased, the permeability deformation of Class G cement sample increased.

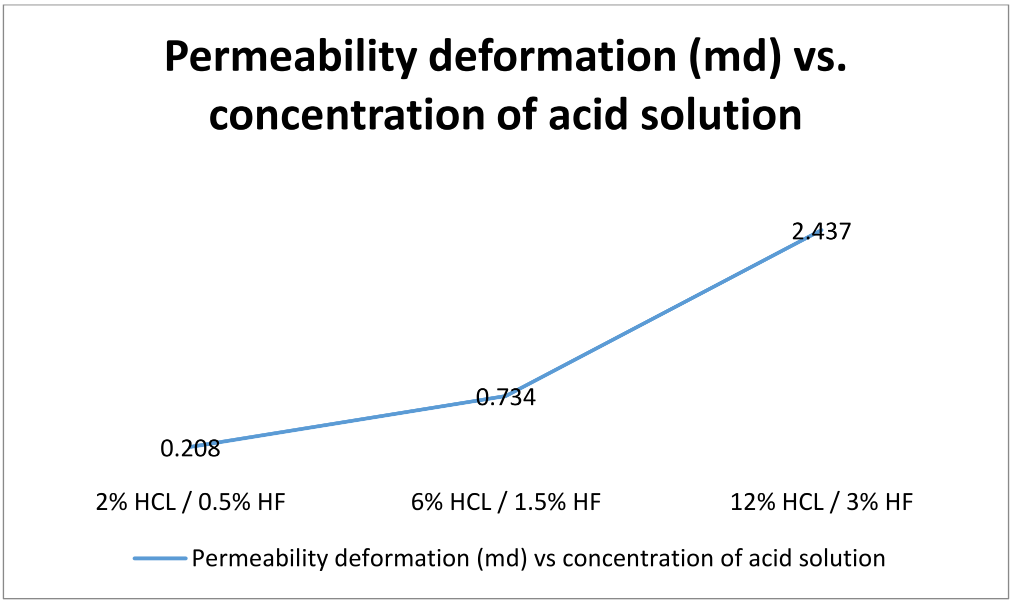


FIGURE 4.4 Permeability deformation (md) vs. concentration of acid solution

It can be seen in the **FIGURE 4.4**, the permeability of Class G cement increased when immersed into high concentration of acid solution. This is due to the amount of H^+ per unit volume in the solution that can be donated or reacted which results in greater reaction between acid and cement.

This can be explained why the acid is a corrosive substance where it can destroy and damage other substances with which it comes into contact. In acidizing treatment, acid used to stimulate the producing formation and it came into contact with the cement casing. In a long term, the cement casing could be damage due to acid attack.

The result showed based on 40 minutes of immersion of cement sample in acid solutions. It was average spending time of acid at the formation before it pumped back to surface. The permeability of Class G cement might be greater if it left immersed in acid solution longer than 40 minutes.

Referring to the past experience that happen in Prudhoe Bay field operating area, it showed more than 37% of primary cementing jobs developed zonal isolation problems after acidizing and 73% of squeeze cement jobs break down after the HF/HCL acid stimulation [14]. One of the reasons why these problems happen might be because of the integrity of well cementing itself. The acid used in acidizing treatment affected the cement.

4.4 SUMMARY OF RESULTS

- Permeameter and Porosity Meter (POROPERM) results proved that the acid used in acidizing treatment affected the permeability of Class G cement.
- Higher concentration of acid solution resulted higher permeability changes of Class G cement.
- Higher concentration of acid solution contributed higher mass loss compared to lower concentration.
- Presence of white milky substance which was known as Calcium Fluorite (CaF_2) and Calcium Chloride ($CaCl_2$) showed acid solution react with calcium of the cement.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSIONS

The objective of this project was to investigate the permeability deformation of Class G cement during acidizing treatment under the reservoir condition which is high pressure and high temperature. Based on the results as stated before, it can be concluded that:

1. Reaction between Class G cement and mud acid produced precipitate such as Calcium Chloride ($CaCl_2$) and Calcium Fluorite (CaF_2).
2. The mass loss of Class G cement when immersed into mud acid was increased when concentration of acid increased.
3. Permeability of Class G cement deformed during acidizing treatment and it was depend with concentration of acid solution. The higher the acid concentration, the higher the permeability of Class G cement deformed.

With these conclusions, the project was done successfully and aided directly or indirectly to worldwide oil and gas industry development.

5.2 RECOMMENDATIONS

Below are the recommendations for future research work:

1. Study the microstructure and depth of penetration of acid before and after immersed into difference concentration of acid solution and with longer time immersion of samples into acid solution.
2. Study the limitation of permeability of Class G cement can deform in a wellbore and propose a few methods to overcome the problem with experimental simulation.
3. Study the effect of different curing condition on the resistance of Class G cement sample toward acid upon acid stimulation.

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APPENDICES

APPENDIX A

Procedure for mixing cement slurries

1. Weigh the amount of cement and water needed.

2. Pour the appropriate amount of water into the mixer container.

3. Turn the power on.

4. Press “Mix 1” switch until it click into position.

5. Place the “fixed/variable switch in the “fixed” position.

6. Press the “start/reset” button to start the motor and begin the timer countdown.

7. Add cement powder to the water.

8. After the cement has been added, place the cover on the mixer container.

9. When the timer countdown reaches 35 seconds, press the “mix 2” button and mix on high speed (typically 12000 rpm) for 35 seconds. When the timer reaches zero, the motor will stop automatically.

APPENDIX B

Start of Operation

1. Close all the valves on the front panel.

2. Turn the POWER switch to the ON position. This supplies power to the entire instrument.

3. Program the temperature ramp and soak parameters into the temperature controller.

4. Assemble each pair of mold bodies. The inside of the molds shall be lightly greased as necessary. Place a mold body on the mold bottom and fill with cement slurry. Place a cover on each mold with the slotted side down. Repeat for remaining mold sets, stacking the molds as they are filled.

5. Clamp the molds together using the threaded rods provided. The small angle brackets should be attached to the threaded rod so the molds may be moved with the wire bail.

6. Lower the filled molds into the stainless steel mold bucket.

7. Lower the bucket full of molds into the pressure vessel.

8. Ensure the cylinder plug threads are thoroughly lubricated. Thread the plug into the cylinder and tighten securely by hand.

9. Tighten the set screws in the plug according to the figure below. The screws must be tightened in the order shown and in three (3) torque increments of 15, 30 and 40 ft-lbs.

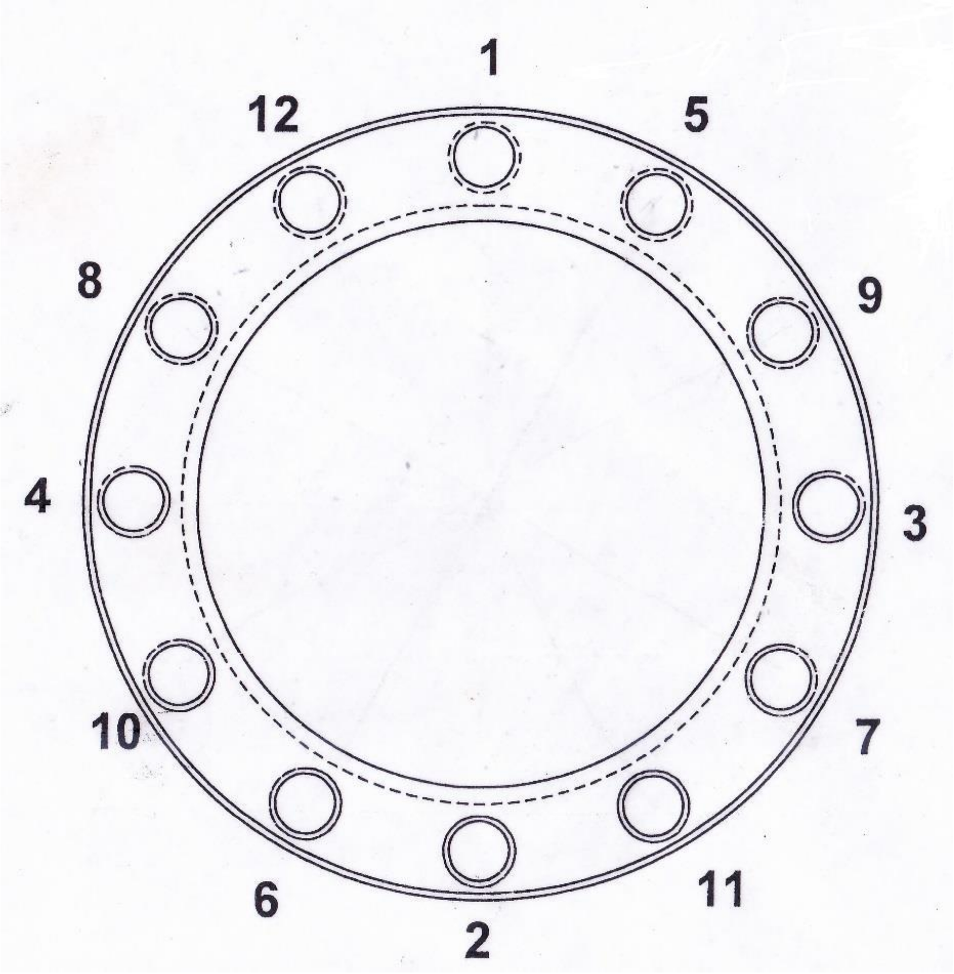

FIGURE B-1 Screw sequence

10. Insert the thermocouple into the opening in the center of the cylinder plug, but do not tighten completely. The air will be vented through the thermocouple opening as the cylinder fills. Be sure to plug the thermocouple into the receptacle on the front panel.

11. Fill the pressure vessel with oil as follows. With all valves closed, open the AIR SUPPLY valve. This valve applies air pressure to the top of the oil reservoir and forces oil into pressure vessel. As the pressure vessel fills with oil, air will be exhausted from the pressure cylinder at the thermocouple connection. A hissing sound should be heard. As soon as the hissing sound stops and oil appears, tighten the thermocouple gland securely. Do not tighten the thermocouple before oil appears or air may be trapped in the vessel preventing pressurization.

12. Adjust the pressure vessel as desired for the start of the test. This is accomplished by placing the PUMP switch in the ON position until the desired pressure is reached.

13. Turn the HEATER switch to the ON position and begin running the pre-programmed temperature profile.

14. Turn the TIMER switch to the ON position.

CAUTION: Top of the curing chamber may become extremely hot. Severe burns can result from touching the pressure vessel or plug.

End of Operation

1. Turn the HEATER switch to the OFF position.

2. Press the run/start key on the temperature controller to stop the program.

3. Slowly open the CYLINDER COOLING valve to start the flow of the cooling water through the internal cooling coils.

4. When the cylinder and plug have cooled sufficiently, open the PRESSURE RELEASE valve.

5. With all the pressure released form the cylinder and the PRESSURE RELEASE valve open, close the AIR SUPPLY valve, open the AIR EXHAUST valve and open the AIR TO CYLINDER valve. This transfers the oil from the pressure vessel to the oil reservoir. The RESERVOR COOLING valve may be open to rapidly cool the oil in the reservoir. A hissing sound will be heard when the pressure vessel is empty.

6. Unscrew the thermocouple in the cylinder plug and remove.

7. Loosen the screws on the cylinder plug.

8. Unscrew the cylinder plug and remove.

9. Remove the mold bucket and molds from the pressure vessel.

APPENDIX C

Procedure of Permeability Measurement

BEFORE OPERATION: Make sure the machine is in good condition and SAFETY FIRST

OPERATIONAL INSTRUCTION:

1. Turn On the computer and run PoropermV1.3b82 file at desktop.
2. Switch on Weigh Balance and Turn On all gas valves connected to the system.
3. From the equipment software select the “Measure” panel.
4. Fill the Sample ID, Diameter, Length, and Weight for each sample you want to measure.
5. Choose the measures you want to do on each sample: Porosity, Permeability or both. By default, we select it both.
6. In case for a sample you only want to perform a Permeability measure you have to fill the sample Pore Volume.
7. Load the core sample inside the core holder.
8. Turn the Confining Pressure valve anticlockwise to pressurize the core holder.
9. Then start the measuring sequence by clicking on the button “Start” at the bottom right side.
10. Wait the first dialog box appears which is asking you if the core sample inside the core holder and click “Yes”.
11. Your samples are finish measured when progress box at bottom of screen stated “Standby”.
12. Select “Calcul” panel to get your measure result.
13. Turn the confining Pressure valve clockwise to vent the core holder.
14. Unload the core sample from the core holder.

SHUTDOWN PROCEDURE

1. Shut down the computer system.
2. Turn OFF the weight balance.
3. Shut OFF all gas valves connected to the system.
4. Turn the main power OFF on the machine.
5. Switch OFF the main power supply.