PERFORMANCE OF DIFFERENT ACIDS WITH VARIOUS FORMULATIONS FOR SANDSTONE FORMATION (PANGKOR SAND)

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the requirements for the
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(Petroleum Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the

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Approved by,

____

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

January 2012

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 by unspecified sources or persons.

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(SITI NOR DAHLIAWATI ZULKEFLI)

ABSTRACT

This project is about the performance of different acids with various formulations for sandstone formation. The objective of this project is to find the most suitable acid formulation for sandstone formation. Sands from Pangkor Island are used as sample for experiment.

Matrix acidizing is done to remove damage near wellbore area by dissolving unwanted or blocking material inside the pore. This operation can be very beneficial if proper planning and appropriate acid formulation is used. (Acidizing Oil and Gas Reservoir:Current Practice and Applications of the Arcasolve Acidizing Process)

To find the most suitable acid for sandstone formation experimental work must be done. After obtain sample from Pangkor Island, the sand grains are converted into sand compacted core to measure porosity and permeability before and after acid treatment is made. The change in porosity and permeability will be tabulated then the result will be observe and analyze. Trial and error method will be used to gain the most suitable acid formulation for sandstone.

ACKNOWLEDGEMENT

In the name of Allah, the Most Gracious, the Most Merciful. Praise to Him the Almighty that His blessing and guidance in giving me strength, courage, patience, and perseverance to endure this 2 semester for Final Year Project.

I am thankful to my parents, my sibling for understanding why I am always not available and not able to go back very often this semester just to finish my Final Year Project this semester. To my sisters who understand why am I not there for her final wedding preparation.

Secondly, I would like to express my deepest gratitude to my supervisor AP Aung Kyaw for helping me giving advice during project process. He also advice me what to do and what to search as well as giving good grade for his students

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ABBREVIATION AND NOMENCLATURE

HBF_4	: Fluoboric acid
HCL	: Hydrochloric acid
HF	: Hydrofluoric acid
$K_{\infty i/\infty f}$	: Initial/final corrected permeability
PVC	: Polyvinyl Chloride
SEM	: Scanning Electron Micrograph
UTP	: Universiti Teknologi PETRONAS
$\Phi_{i/f}$	: Initial/final porosity
µm	: Micron meter ($\times 10^{-6}$)

CHAPTER 1

INTRODUCTION

1.1. Background Study

The purpose of matrix acidizing is to treat damage by workover and stimulation practice to increase production. A lot of advance method has been patented nowadays to improve acidizing process. There several number of acid that can be used for matrix acidizing. Some of them are conventional acid such as hydrochloric acid, hydrofluoric acid and acetic acid. Some other acids are mud acids and organic acid which are invented by combining different chemical together producing new acids.

Different formation may need different acid for acidizing purposes. This is because acid use for matrix acidizing is based on the damage that has happened or the physical properties of the rock or formation itself.

This study is about finding the most suitable acid formulation to react with sandstone formation. This study will be done experimentally using sand sample from Pangkor Island. Acidizing can be used to removing damage during drilling or completion processes.

1.2. Problem Statement

There are a lot of acidizing method using various acid formulation has been used nowadays. The challenge is to find the most suitable acid for sand sample coming from the Pangkor sand. Every region or basin formation may need different acid formulation because of the physical properties that is different. Thus to find the most suitable acid formation experimentation must be done.

If the acidizing process are using suitable acid it could be very beneficial and low cost. Otherwise, serious damage could happen. Some of the things that could happened is when the acid is not suitable, and after it react with the formation it produce precipitates. This precipitates can clog the pore space resulting decrease in porosity and permeability. Acid can easily corrode the casing or other tools that are in operation during acidizing treatment. Thus, tools use must also be taken in considerations before start treatment.

1.3. Objective

The main objective for this research is to find the most suitable acid formulation experimentally for sandstone formation using sand sample from Pangkor sand. The outcome of this experiment will be record for analysis.

1.4. Scope of study

This study is for finding the most suitable acid for sandstone using laboratory approach. Acid that is commonly used in the industry for pre-flush, main flush and after flush must be reviewed before starting the experimental work. The expected result for this project is the acid that can increase the highest porosity and permeability of the sample.

Research on different type of acid such as conventional acid, mud acid and organic acid must be done. From this research a list of acid will be made and the acids will be requested for experimental purposes.

Sandstone is very sensitive to injection fluids. The acids use must depend on the formation for example the percentage of carbonate, clay, tool used, cementing material and etc.

CHAPTER 2

LITERATURE REVIEW

Matrix acidizing is as old as the oil industry itself. In 1896, acidize limestone using hydrochloric acid has been patented. As for sandstone, acidizing using hydrofluoric acid was patented in 1933(Crowe C.). Matrix acid job is a low budget operation. Thus, it must be plan carefully to make it a success. Otherwise, if it's not the damage repair could cost a lot.

Acidizing processes is used to increase production. It is commonly used for work over and stimulation practice in the oil and gas industry. This process can help stimulate the true permeability of sandstone formation. The fluids are pumped into the porosity of the rock at below the fracturing pressure and the acid reacts with a large portion of the formation.

Different acids are used in conventional acidizing treatment such as hydrofluoric acid, hydrochloric acid, acetic acid and much more. The majority of acidizing treatment carried out utilized hydrochloric acid (HCL). However the very fast reaction rate of hydrochloric acid and other acid can limit their effectiveness. This means the acid does not penetrate very far into the formation before it is spends.

There are a number of ways to slow the acidizing process. Some of the methods that have been developed are emulsifying the aqueous acid solution in oil to produce an emulsion, dissolving the acids in non-aqueous solvent and the use of non-aqueous solutions of organic chemical which release acid when in contact with water. The use of methyl acetate which hydrolyses slowly at very high temperature can produce acetic acid. Applying the same hydrolyses approach, Fluoboric acid can turn to hydrofluoric acid in-situ. In addition, retarding the acid can be achieved by gelling the acid or oil wetting the formation solids.

Sandstone acidizing consist of three stages. First stage is pre flush followed by main flush and finish with post flush. Pre flush is to displace the existence salt water and to dissolve carbonates. Main flush or mud acid stage is to dissolve clay, feldspar or any other material near wellbore area. Final stage is after flush which is to displace the mud acid stage (J.L Gidley, 1996). Below is the example of acids used for two stage job:

Pumping Schedule for a Two-Stage Job						
		Step	Fluid	Volume bbl	Flow rate bbl/min	Time min
Stage 1	1	Preflush	HCl 15%	17.3	2.2	7.9
	2	Main fluid	RMA 13/3[1]	68.2	2.2	31.0
	3	Overflush	HCl 4%	33.0	2.4	13.8
	4	Overflush	HCl 4%	20.7	4.8	4.3
	5	Diverter slug	HCl 4%	3.1	4.8	0.6
	6	Preflush	J237A[2]	17.3	4.8	3.6
Stage 2	7	Main fluid	HCl 15%	12.6	4.8	2.6
	8	Main fluid	RMA 13/3[1]	55.6	1.1	50.5
	9	Overflush	RMA 13/3[1]	53.7	1.1	48.8
	10	Tubing displ.	NH_4Cl brine 3%	33.0	1.2	27.5

1. Regular Mud Acid, 13% HCl, 3% HF. 2. Four-micron particulate oil-soluble resin, usable up to 200°F.

Figure 1: List of acid use for two-stage job.

Acidizing job can be even more efficient if what material blocking the pore can be determined. This can be resolved by using Scanning Electron Micrograph (SEM) tool. SEM can help ones determine which acid can dissolve which particle or element. Below is the example of the micrograph before and after applying mud acids and Fluoboric acids;

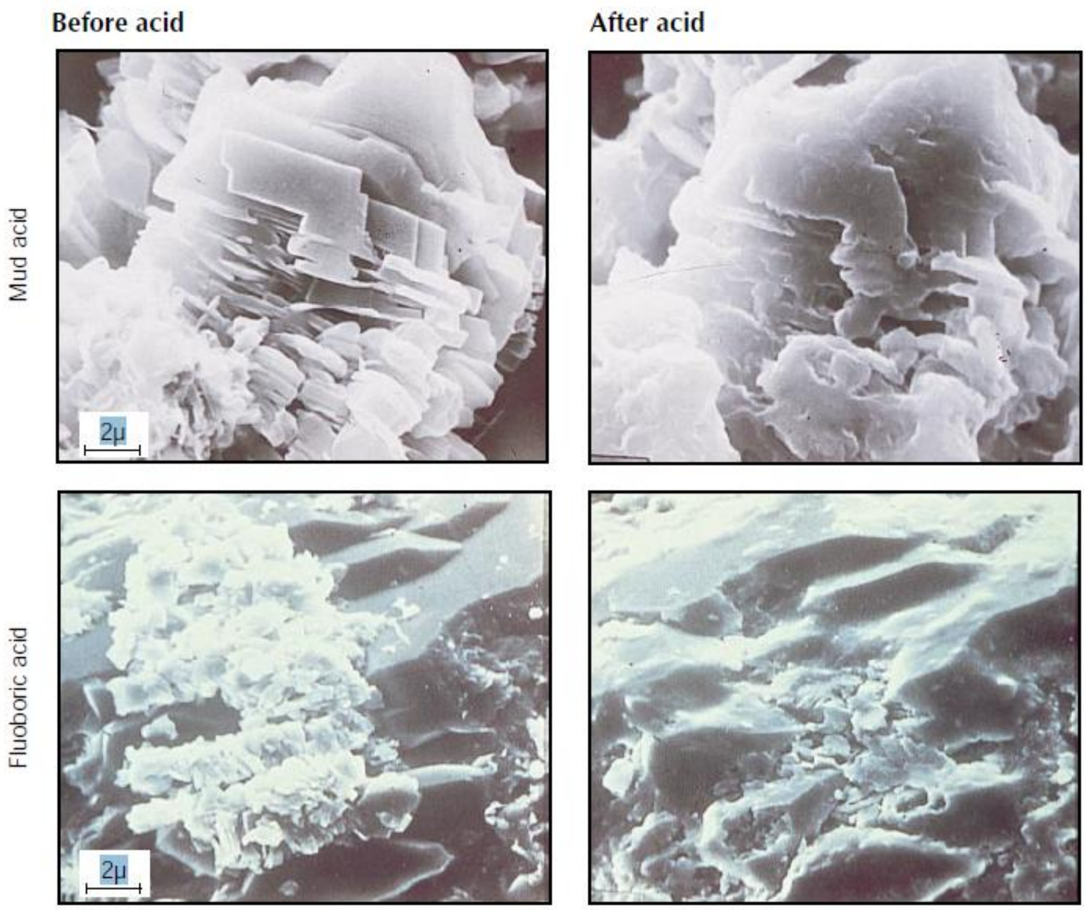


Figure 2: Micrograph of sample before and after applying acids.

There are popular perceptions that acid stimulation have high rate of failure. In reality there are limited numbers of reasons or controllable causes for sandstone acidizing treatment failure, and all can be avoided with proper treatment planning and executions.

Industry expert such as Harry Mcleod and George King have indentified and listing the common causes of sandstone acidizing treatment failure. As it turn out, most acidizing treatment failure can be one or more of the following;

1. Treating a well that has high skin, but no damage
2. Using acid on formation that was not adequately perforated
3. Using the wrong type of acid to remove damage
4. Using improper acid volumes and/or acid concentration for formation mineralogy
5. Using dirty water to mix preflush or overflush stages
6. Failure to clean acid or water tanks
7. Additives overuse or misuse
8. Pumping the acid job above fracturing pressure (with exceptions)
9. Shutting in the acid treatment too long before producing back.

Understanding that the most treatment failures are due to one or more of these reasons simplify the process. It also can ease the mind when sandstone acidizing treatment design considerations seem hopelessly complicated.

CHAPTER 3

METHODOLOGY

This project will base on laboratory works. After doing some research on common acid used for acidizing process, acids listed was requested for experimental use. Sample used for this project are coming from Pangkor Island. The basic flow of this project is presented as follow;

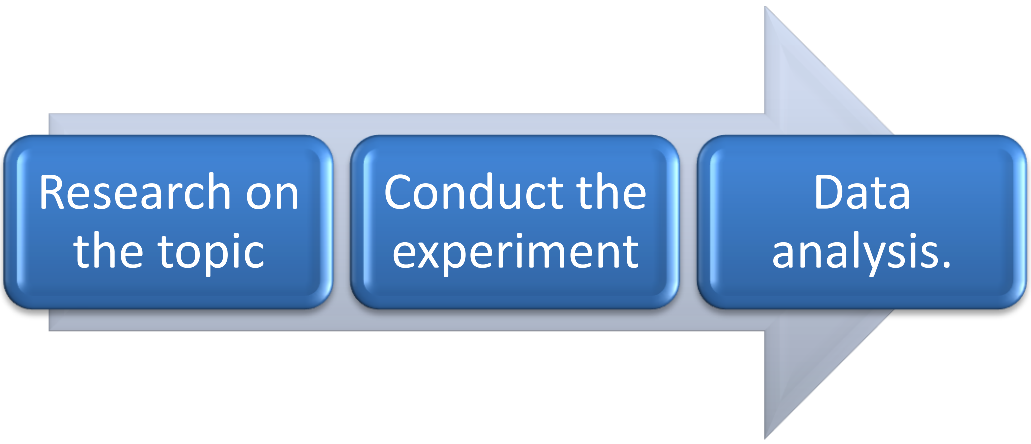


Figure 3: Project flow

The experiment was conduct in UTP during Final Year Project II (FYP II) period. The Gantt chart for this project is showing the timeline that has been used for this project. The gantt chart is presented in the next page.

No	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Topic Selection / Proposal							SEMESTER BREAK							
2	Research Work - Research paper														
3	Submission of Proposal Defense Report						●								
4	Proposal Defense (Oral Presentation)									●					
5	Project Work Continues; - Request acids - Taking samples														
6	Submission of Interim Draft Report												●		
7	Submission of Interim Report													●	

Table 1: Gantt chart for FYP1

No	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Project Work Continues - Experiment							SEMESTER BREAK								
2	Submission of Progress Report								●							
3	Project Work Continues - Data analysis															
4	Pre-EDX											●				
5	Submission of Draft Report												●			
6	Submission of Dissertation (soft bound)													●		
7	Submission of Technical Paper													●		
8	Oral Presentation														●	
9	Submission of Project Dissertation (Hard Bound)															●

Table 2: Gantt chart for FYP2

There are a total of nine samples saturated with nine different acids combination to observe the change in permeability and porosity. The process and experimental procedure is explained below.

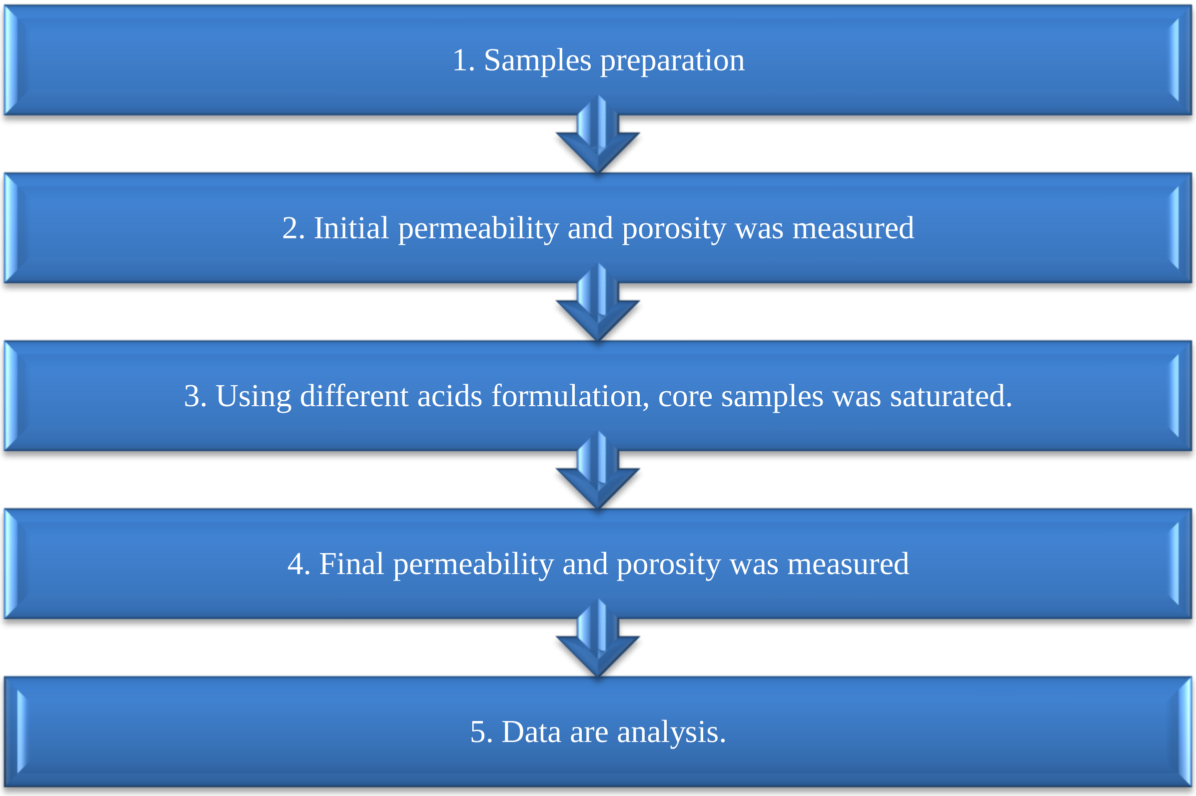


Figure 4: Experimental Procedure.

First procedure is to prepare the sample. Sample preparations start from taking the sand grain from Pangkor Island to cylindrical shape core sample. The sand grain was taken earlier in January by digging the formation a feet deep and 100m from the shore. After successfully transfer the sand back to UTP, the sand grain is then wash and dry under the sun for one whole day before proceed to next process.

Next process is to sieve the sample. Sieving size used was 1.18mm, 710µm, 600µm, 425µm, 300µm and 150µm. Sieving process took 10 minute each run. As a result, most of the sand was retained in less than 300µm size. Because of that, the size of grain chosen to form the core sample is less than 300µm.

Figure 5: sieving equipment

In order to form sand grain into core sample, 22% of epoxy resin with hardener was mixed into the sand grain and compressed naturally. The mixture was left in 1.5 inch PVC pipe under the sun to make sure it is fully dried. Unfortunately, the PVC pipe was extended to 2 inch diameter. The equipment to measure porosity and permeability can only be use with 1 or 1.5 inch diameter cylindrical core sample. Thus all the samples are partially submerge in the cement. By using 1 inch drill bit, coring has been done using coring machine. The dimension of all nine samples is 1 inch diameter with 2 inch height.

Figure 6: Core sample preparation

Nine samples has been prepared, the initial permeability and porosity has to be measure before saturate with acids. After acids saturation the permeability and porosity was measured again thus the change of these two parameters can be observed and the data analysis can be done. These parameters can be measure using POROPERM equipment.

The POROPERM instrument is a permeameter and porosimeter used to determine properties of plug sized core samples at 300 psi confining pressure. In addition to the direct properties measurement, the instrument offers reporting and calculation facilities using window operated software. The measurement is based on unsteady state method (pressure falloff) whereas the pore volume is determined using Boyle’s law technique.

Figure 7: POROPERM equipment

All nine core samples are saturated in nine different acids combination. Acid saturation has been use instead of injecting acid into the core sample because the equipment available is easily corroded to acid. Steel is easily corroded to hydrochloric acid and glass is corroded to hydrofluoric acid. All the equipment used was plastic ware to prevent dysfunctional of the equipment. The acid combination that has been use is listed in table below.

No of core	Acids combination
1	12%HCL+3%HF
2	12%HCL+4%HF
3	12%HCL+5%HF
4	12%HCL+6%HF
5	12%HCL+8%HF
6	15%HCL+2%HF
7	15%HCL+3%HF
8	15%HCL+5%HF
9	HBF_4

Table 3: list of acids combination

Acid saturation took 4 hour each run. This experiment has to be done in the fume hood to avoid the fume evaporate to the air and become hazard to others. To speed up the process, desiccator with vacuum pump was used to make sure the core sample was fully saturated with acid. After this process the core sample was dry up in the oven and ready for final measurement. Lastly, SEM was done to help understand the behavior of the samples properties followed by data analysis.

Figure 8: Desiccators with vacuum pump.

CHAPTER 4

RESULT AND DISCUSSION

As mention before, the objective of this project is to find the most suitable acid combination for Pangkor formation (Pangkor sand). The suitable acid combination can be determined by finding the most effective and appropriate permeability and porosity change before and after acid saturation has been made. The expected result is to find the most increase in permeability and porosity. SEM has also been run to help understand the sample behavior after saturated with acids.

The result will be separated into two parts which are;

1. Acid saturation results
2. SEM results.

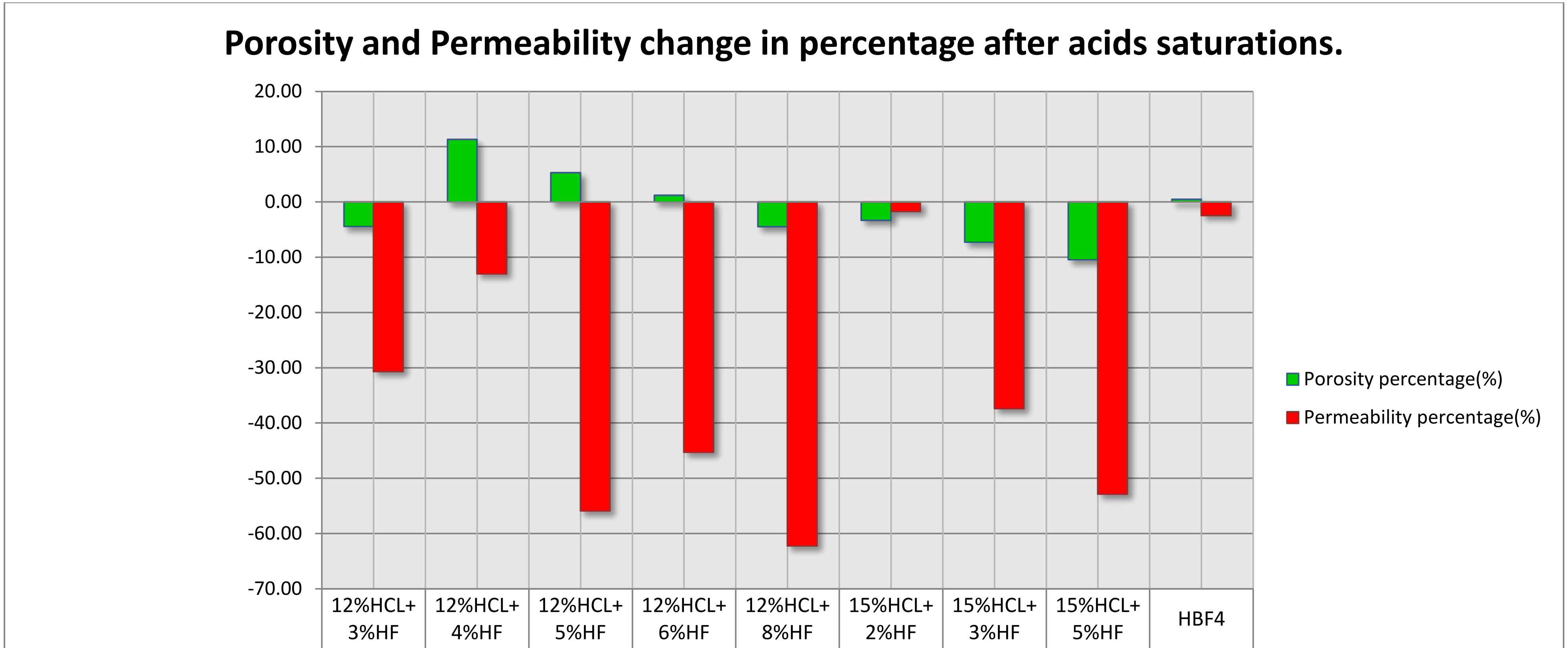
4.1 Acid Saturation Results.

In table below show the result for nine samples with respective acid combination used. Φ is the porosity that has been measured. The initial and final porosity are shown below. K_∞ is the corrected permeability that is calculated by the equipment which is POROPERM. The percentage of difference for permeability and porosity was calculated and the result shown in the last 2 column in table below.

Based on experimental result, three samples only increase in porosity but decrease in permeability, five samples decrease in both permeability and porosity and only one sample with almost no reaction as the change is too small.

CORE NUMBER	ACID FORMULATION	DIAMETER (mm)	LENGTH (mm)	WEIGHT$_I$ (g)	WEIGHT$_F$ (g)	Φ_i (%)	Φ_f (%)	$K_{\infty I}$ (mD)	$K_{\infty f}$ (mD)	Change in Φ (%)	Change in K_∞ (%)
1	12%HCL+3%HF	25.2	51.1	38.34	35.50	26.120	24.954	19,857.00	13,762.05	-4.46	-30.69
2	12%HCL+4%HF	25.2	50.4	42.75	40.68	17.390	19.353	1,487.00	1,293.19	11.29	-13.03
3	12%HCL+5%HF	25.2	51.2	40.05	37.61	24.607	25.907	11,867.50	5,232.19	5.28	-55.91
4	12%HCL+6%HF	25.2	51.0	38.48	35.89	26.920	27.24	22,920.00	12,532.58	1.19	-45.32
5	12%HCL+8%HF	25.2	51.4	38.37	36.69	26.980	25.767	23,820.00	8,989.26	-4.50	-62.26
6	15%HCL+2%HF	25.2	51.0	44.77	43.34	11.840	11.447	20.30	19.944	-3.32	-1.74
7	15%HCL+3%HF	25.2	51.2	43.57	42.26	14.592	13.53	913.57	571.589	-7.28	-37.43
8	15%HCL+5%HF	25.2	51.1	40.98	40.03	21.217	18.997	6,720.00	3,165.32	-10.46	-52.90
9	HBF_4	25.2	51.0	43.98	42.75	13.937	14	379.34	369.94	0.45	-2.48

Table 4: Acids saturation result



	12%HCL+ 3%HF	12%HCL+ 4%HF	12%HCL+ 5%HF	12%HCL+ 6%HF	12%HCL+ 8%HF	15%HCL+ 2%HF	15%HCL+ 3%HF	15%HCL+ 5%HF	HBF4
Porosity percentage(%)	-4.46	11.29	5.28	1.19	-4.50	-3.32	-7.28	-10.46	0.45
Permeability percentage(%)	-30.69	-13.03	-55.91	-45.32	-62.26	-1.74	-37.43	-52.90	-2.48

Graph 1: Acid saturation result

The permeability for most of the samples is too good to be true. The weirdness of this value may come from machine and human error. The machine may have given wrong value as the gas input was originally wrong and it may affect the calibration of the equipment. The old part of the equipment such as rubber sleeve inside the tools may contribute to the error as it may not be as tight as before. This equipment also tends to give result similar to previous core that has been tested. To get accurate result, the whole equipment need to be restarted and it cannot be used for a long time.

As for human error, the sample preparation may have effect the property of the sand sample. The epoxy resin that has used as the bonding agent may have affected the permeability of the samples. Core compressions for the entire core are different because it is only done by hand without any appropriate tools. The compression may not be homogenous throughout the core sample. The resin that bind the sand grain is not well compacted thus leaving a lot of pore spaces inside the core sample. All these reasons may contribute to the high permeability of the core samples.

Highest number of sample experience decrease in permeability and porosity. The acid combinations that contribute to this result are 12% HCL + 3%HF, 12% HCL+ 8% HF, 15%HCL + 2% HF, 15% HCL + 3% HF and 15% HCL + 5% HF. There are few possible causes of this phenomenon. Those acids combination may be improper for Pangkor Island formation. When improper acid are used for acidizing treatment, the opposite reaction may occur as happened to this 5 samples. The mineralogy of this sand has not been defined thus the suitable acid for this formation cannot be defined theoretically. Trial and error basis has been used for this experiment resulting such a shocking result.

Hydrochloric and hydrofluoric acids are common acids for sandstone treatment. Improper acid concentration may also be the reason of the acid treatment failure. In here, only 1 acid combination with various concentration used and 5 of the concentration chosen resulting in bad result.

The other reason that may contribute to decrease in permeability and porosity is the mineralogy. Mineralogy for Pangkor sand sample was not defined. This is because of time constraint as the duration for this project is only eight months. The equipment for defining mineral is available but the gas that needed has not been arrived yet. Mineral determination can also be done by doing thin section but the queue for this equipment is quite long thus the opportunities to define mineralogy cannot be obtained. As a result, the suitable acid cannot be defined theoretically because of the reason that the dissolved minerals cannot be known as well as the remaining minerals too. Pangkor sand contained a lot of coral even after dig it a feet deep inside the formation.

Saturation period for acid treatment was too long and that can be one of the reasons. In this experiment the saturation time may have been too long especially for high hydrofluoric acid concentration. This would result in precipitate start to accumulate in that particular place where it has been dissolve. The acid should be kept moving to prevent reprecipitation of acid reaction.

There are 3 samples with increase in porosity but decrease in permeability. One of the reasons is that the acid reaction may result in precipitation. The higher the concentration of hydrofluoric acid, dusty white color fine particles ware discovered on the surface of the beaker after acid saturation process finished. This may be the reason why the permeability is decreasing. It was due to particles clogging up the interconnected pore inside the core samples.

For the ninth sample Fluoboric acid was used to do acid treatment. The acid formulation is shown as follow.

<u>Fast reaction</u>

$$H_3BO_3 + 3\,HF \longrightarrow HBF_3OH + 2H_2O$$

<u>Slow reaction</u>

$$HBF_3OH + HF \longrightarrow HBF_4 + H_2O$$

The permeability and porosity change in this sample is not much different. Permeability change only -2.48% and porosity change only 0.45%. As shown in chemical reaction above, the first part is the fast reaction which has been done for around 4 hours. The second stage is to add hydrofluoric acid into the previous solution. This stage is slow reaction stage which usually takes a long time but this experiment only conducted around 17 hours.

Some chemical analysts say that slow reaction could take from a day or two to few days. They used the help of NMR tools to identify when the reaction is finished. Here the equipment is not available thus the solution was left stirring over the night. The acid is believed to be not fully developed when it was used to saturate core sample. Solution preparation may not be complete or may be contaminated by other materials along the night. Thus sample number nine in which core saturated with Fluoboric acid is considered failed.

4.2 Scanning Electron Microscope (SEM) result

SEM is a powerful imaging tool that can help capture the sample in clear microscopic image. SEM has been used in this project to get better understanding about the sample behavior after it has been saturated in acid. Few samples have been tested using this equipment. All samples cannot be run because of time constraint and the queue is so long and it can only be booked on 1st day every month.

The SEM image shown in figure 9 and 10 below is from original sample to sample that has been saturated with 12% HCL + 8% HF. This image is in 100X magnification from its original size. This combination resulting permeability change is -62.26% and -4.5% change in porosity. There are slightly visible changes in these two image as it clearly state that the acid combinations seems to be failed to dissolve the sand sample.

The difference between figure 11 and figure 12 shows difference between original core samples and sample after saturated with Fluoboric acid respectively. This image is in 100X magnification from its original size. Comparing these two images, the one that has been saturated seems to be dissolving but not fully reacted. There are sign of reaction between sand particles with acid. The duration of acid saturation for this sample may not be enough or too short. In this experiment, four hours for each sample has been used. The temperature of the acid was lower than the one used in the industry. In the industry the acid are injecting in the borehole to the formation and as well known deep formation preserved high temperature. In this experiment, room temperature has been used for all samples saturate in all acids combinations. More time or high temperature could be the answer to successfully increase the permeability and porosity for this particular acid combination. Mineralogy investigation should also need to be done in order that the appropriate acid combinations can be determined to dissolve the unwanted particles.

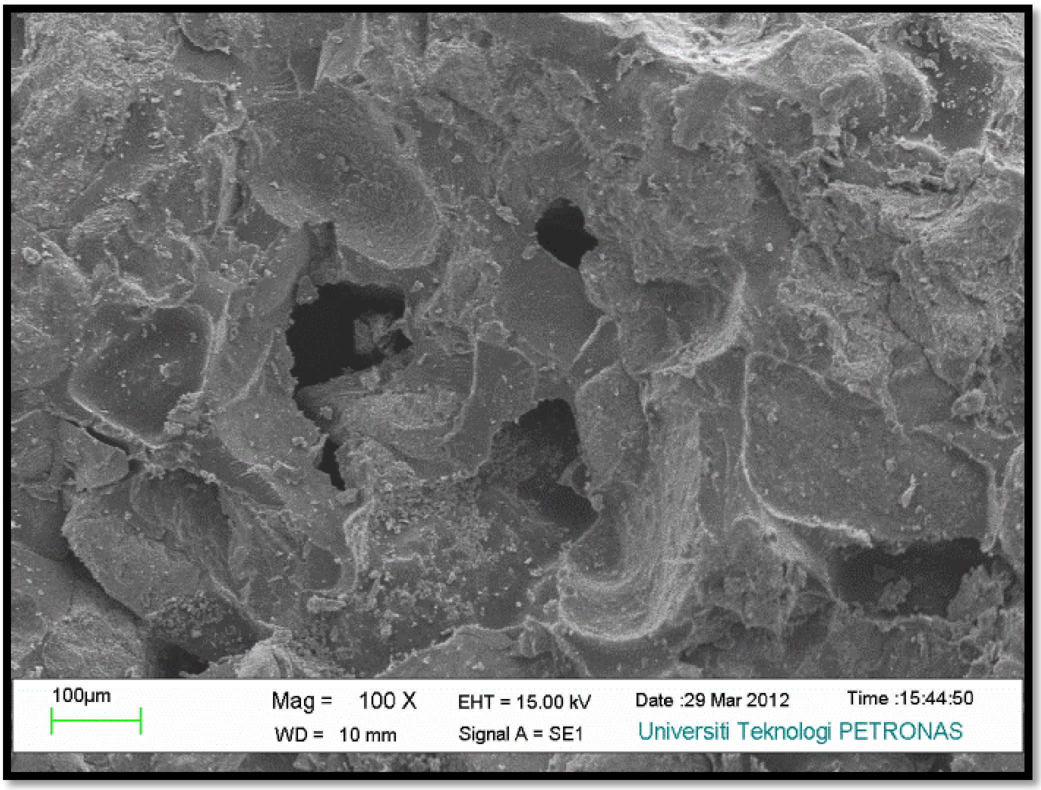


Figure 9: Original core sample

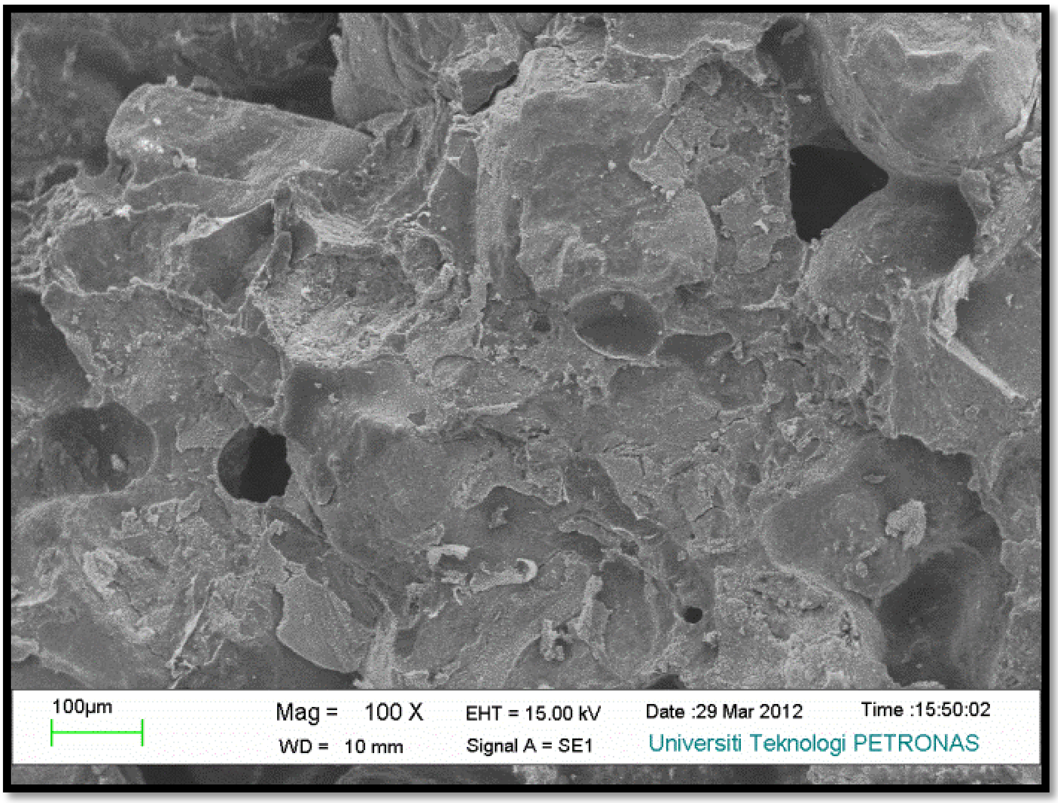


Figure 10: Sample after saturated with 12% HCL + 8% HF

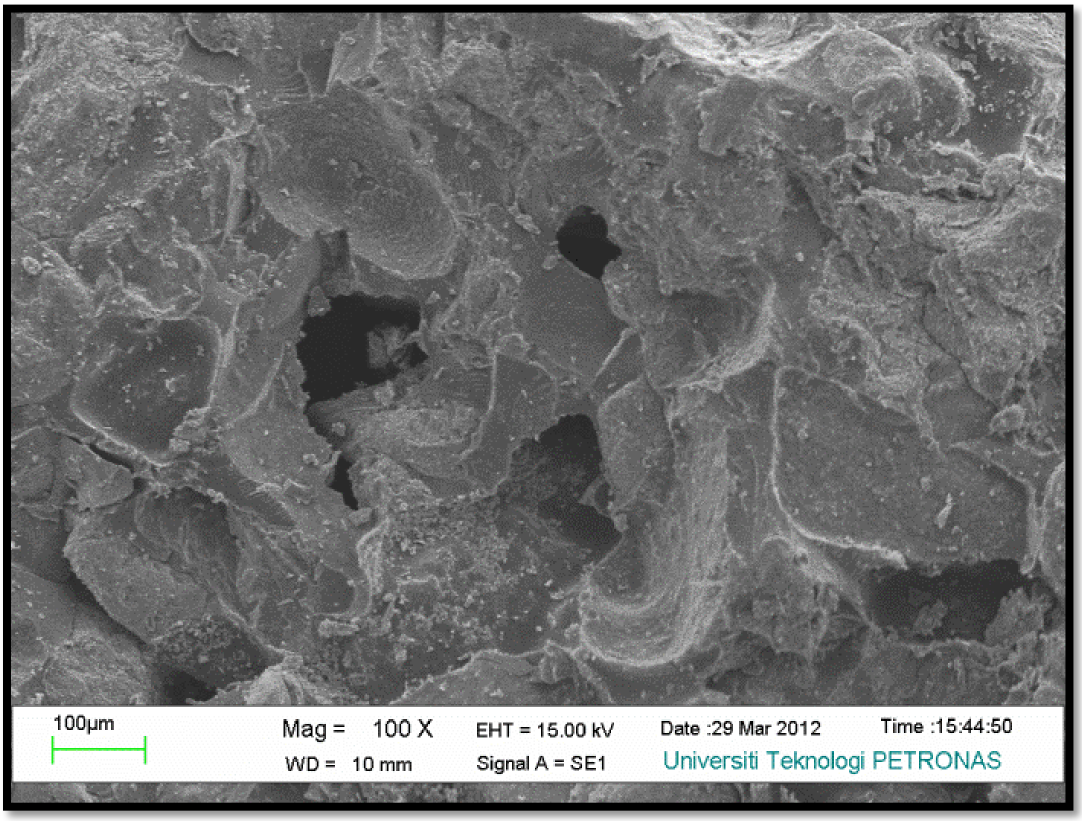


Figure 11: Original core sample (2)

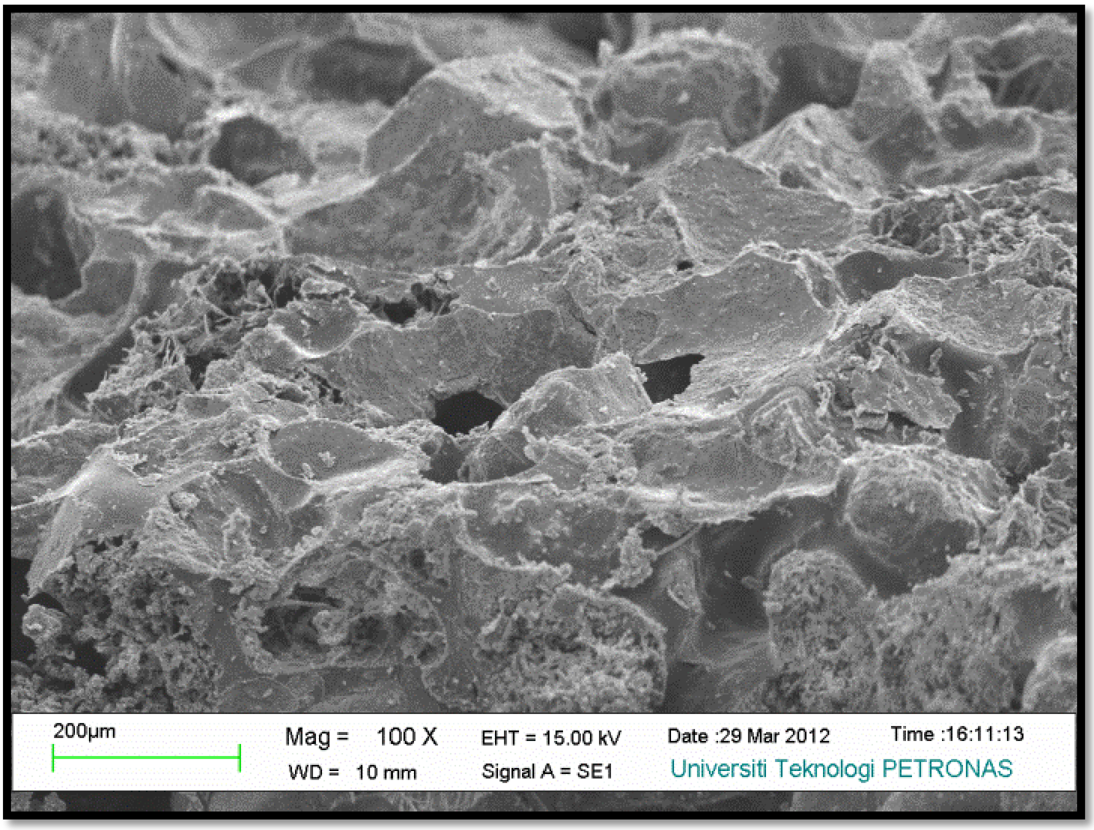


Figure 12: Sample after saturated with HBF_4

CHAPTER 5

CONCLUSION AND RECOMMENDATION

The objective for this project is to find the most suitable acid formulation or combination for sandstone formation coming from Pangkor Island using laboratory approach. This project has been through a lot of obstacle to be completed less than seven months.

The permeability reading used in this project is too good to be true and the result gain from the experiment was also unpredictable. Most numbers in this project is a good number but in the end out of nine samples with nine different acid combinations there is not one of that sample succeeds to meet the objective.

As a conclusion, in the author’s opinion, hydrochloric and hydrofluoric acid combination is not suitable for samples that have been prepared using Pangkor outcrop sand. The conclusion may differ if further study in this project is made in the future. Mineral investigation is crucial for this study to understand the reaction between acid and mineral. Unfortunately, the mineralogy identification cannot be done due to unavailability of the equipment.

This study can be pursuing more in details if time provided are longer. If the time allocated is long enough, mineralogy can de identified. Mineralogy can help to understand which mineral component from the sand sample dissolve by that particular acid used. If the sand mineralogy are known, the acid for dissolving that particular minerals are known, acid job can easily be done. The mineralogy has not been defined because the equipment is still under maintenance after almost a semester and to do thin section it would take more than a semester.

After almost 7 months doing this project, what that has been seen is that the equipment in petroleum and geosciences faculty is limited. Only one apparatus for investigating each properties is available may be because the equipment cost is very high. Another point is the availability of the equipment. A lot of equipment ware under maintenance for quite long time. The technician was not always available and sometimes to repair the equipment they have to call the manufacturer. This will result in long queue which will affect our timeline for Final Year Project or other kind of projects.

There are a lot of human errors in this project. One of the recommendations to reduce the error is to use different binding agent for the samples. Epoxy resin may not be the most suitable one but it was used because it is the only one that is available at that time.

The other suggestion is to compress the sand using the compressing unit. There might be suitable compressing unit available for this purpose. Compression applied to all the samples should be uniforms thus the porosity and permeability values may be consistent.

If there is a core making available nowadays, this project may be an easy task. This is because the sample preparation part is one of the most challenging parts in this process. No proper equipment was available and thus the sample properties are far different compared to sand core sample taken from the field.

There are plenty of rooms for improvement for this project. All of the procedure for this experiment is combination of research and ideas from lecturer and laboratory technologies. Last but not least, to conclude, this experiment is success.

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