EXPERIMENTAL STUDY ON PERMEABILITY REDUCTION DURING CARBON DIOXIDE (CO_2) INJECTION DUE TO ASPHALTENE PRECIPITATION IN LIGHT OIL

By

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10793

Dissertation Final Year Project
Submitted to the Faculty of Geoscience and Petroleum Engineering
In partial Fulfillment of the requirements for the
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(Petroleum Engineering)

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CERTIFICATE OF APPROVAL

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Tronoh, Perak Darul Ridzuan

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LIST OF FIGURES

This study will concentrate on experimental analysis through coreflood tests to evaluate the asphaltene precipitation and permeability reduction. The dead Dulang reservoir oil and sandstone rocks are used as porous media during conducting the experiment. A systematic approach is presented for diagnosing the findings in order to verify that oil permeability will be reduced in the present of asphaltene precipitation in light oil reservoir.

1.2 Problem Statement

Reservoirs with even minute asphaltene content are exposed to asphaltene precipitation due to not only pressure depletion during primary recovery but also composition change in fluid during gas injection[4,5,6,7,8,9]. Precipitation of asphaltene could occur in reservoir, wellbore and production facilities, and could cause adverse effect on the oil recovery, operational facilities and rock properties. Often resort must be had to expensive and disruptive remedial measures to encounter asphaltene problems. Then, it is difficult to control and cure the effects of asphaltene precipitation such as permeability reduction and wettability alteration within the reservoir. Therefore, a complete evaluation and comparative studies with proper plan required to reduce asphaltene precipitation problems.

1.3 Objectives of Study

The ultimate purpose of this project is to study the effect of asphaltene precipitation on absolute permeability in light oil during CO_2 flooding. In order to complete the study, following objectives are expected to be achieved:

i. To evaluate permeability reduction during CO_2 flooding.
ii. To conduct coreflood test using different rate of CO_2 injection at constant temperature and pressure.
iii. To measure and compare change in amount of asphaltene content before and after the coreflooding.

important effect of hydrocarbon composition is that mole fraction of resins is always larger than that of asphaltenes and hence the micelles are expected to be richer in resins[16]. **Figure 1** below shows asphaltene-resin micelles.

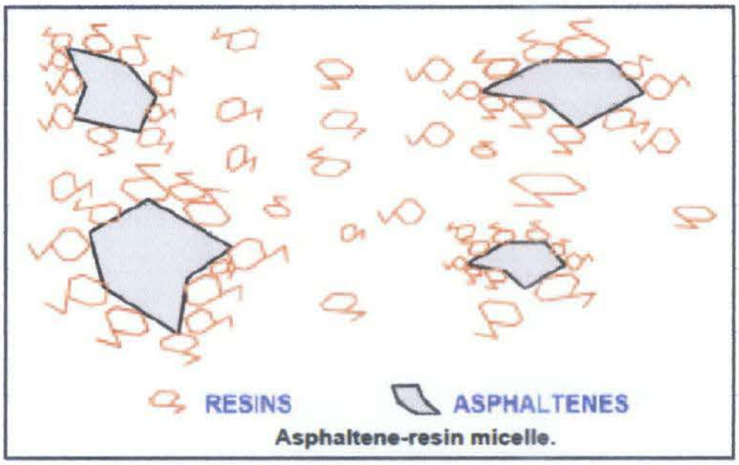

Figure 1: Asphaltene-Resin Micelle[5]

2.2Asphaltene Precipitation and Deposition

Asphaltene precipitation and deposition within the production systems consists of wells, flowlines and surface facilities is far less problematic compare to its precipitation in the pores of reservoir rocks due to ease of access and the possibility of designing remedial treatment. Deposition in the production system can usually be treated relatively easily by chemical or mechanical process[17]. For chemical treatment, a solvent such as Xylene is used to flush the system while a scraper is used to remove deposited sludge for mechanical treatment.

Subsurface rock system deposition is very difficult to treat because not only deposition itself as an interference to flow, but the possibility of blocked pore for delivery path of a particular reservoir. Deposited asphaltenes can reduce the hydrocarbon effective mobility by blocking pore throats thus reducing the rock permeability, adsorbing onto the rock and altering the formation wettability from water-wet to oil-wet, and increasing the hydrocarbon viscosity by forming a colloidal solution in the oil phase[2]. From these phenomena, a significant potential loss of hydrocarbon produced will be generated.

Generally, asphaltene deposition in reservoirs happens when asphaltene flocculate by depressurizing the oil. The second reason for asphaltene deposition is when solvent such as natural gas liquids, natural gas and CO_2 are used to displace oil in EOR. There are other factors that affect the asphaltene precipitation inside a reservoir. These may include the nature, saturation and distribution of reservoir fluids, the mineralogy and properties of the rock, the pressure and temperature, the nature of injection fluids, the electrokinetic effects due to streaming potential generation due to reservoir fluid flow, the asphaltene and resin contents of the reservoir oil and the amount of formation brine and its compostion[18,19,20,21].

From previous studies reported by Fisher and others [22], the process of asphaltene precipitation followed several important steps: (1) The first step is precipitation, this is when the solid particles from a distinct phase as they come out of solution. The quantity and size of solid particles at this stage could be quite small. (2) The second step is the flocculation stage, a process by which the small solid particles clump together and grow larger. (3) The third stage is deposition, a point at which the particles are so large that they can no longer be supported by the liquid and therefore settle out on the solid surfaces. Although asphaltene problems had occurs for last three decades, its mechanism of deposition and precipitation still not fully understood and several questions remain unanswered especially during oil recovery because of its complexity.

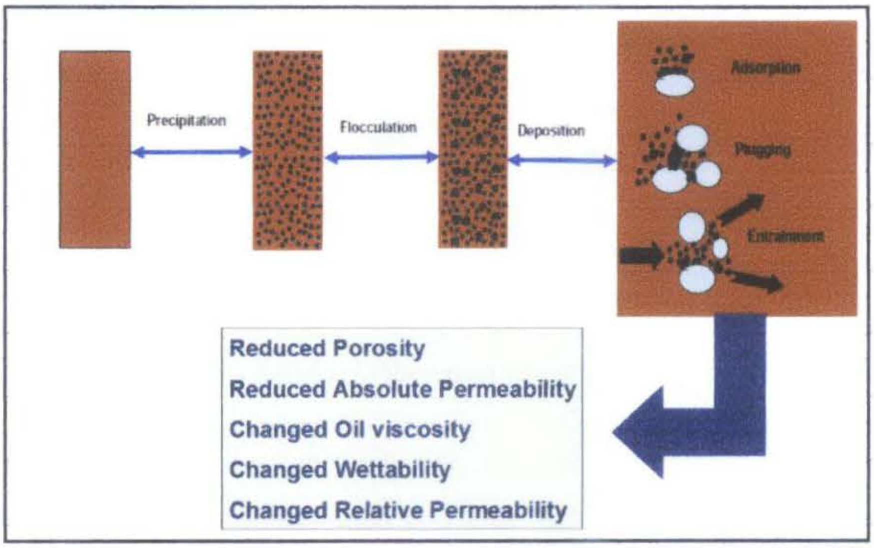

Figure 2: Asphaltene Deposition Mechanism[11]

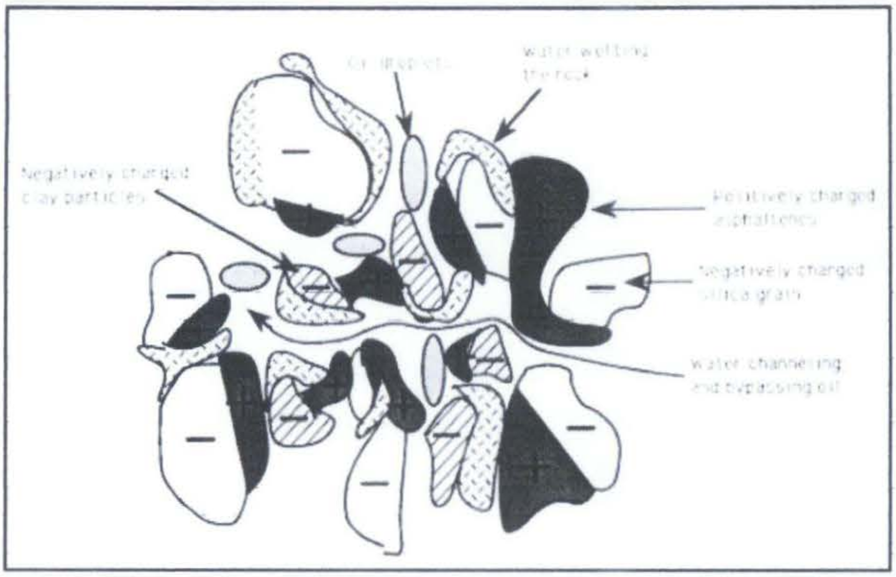

Figure 4: Asphaltene adsorbed on the rock causing wettability changes[2]

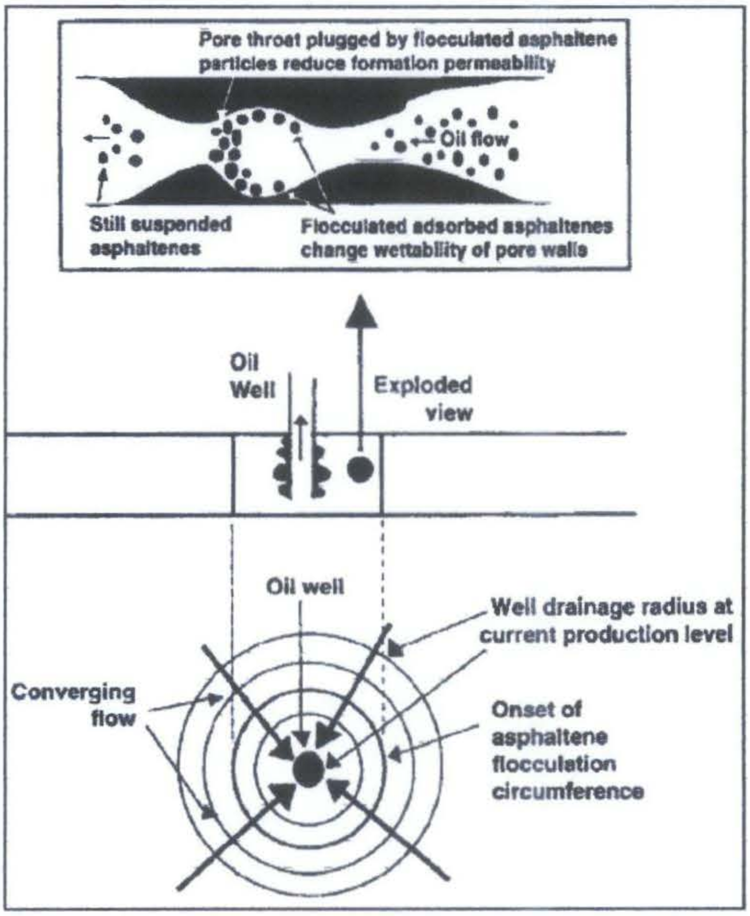

Figure 5: Reservoir Flow Behaviour [2]

In general, CO_2 is soluble in crude oil at reservoir pressure that CO_2 injection into reservoir will increase oil production by re-pressuring old formations and mobilizing the oil[3]. The injected CO_2, when it contacts with reservoir oil can cause changes in the fluid behaviour and equilibrium conditions which favour precipitation of asphaltene[5,24,25]. Initially at early stage, oil and CO_2 mixture has relatively low content of asphaltene during CO_2flooding. As a result, the execution of this CO_2injection will lead to increment of asphaltene precipitation and deposition.

However, it has been observed that amount of the asphaltene content in crude oil is not the reason of asphaltene precipitation. Studies confirmed that asphaltene will precipitate more easily in light oil because the solubility of asphaltene will be decreased compare to heavy oil which has much higher asphaltene content. For instance, the Venezuelan Boscan crude oil with 17.2 wt% asphaltene may not manifest asphaltene problem, whereas crude oil of Hassi-Messaoud with 0.15 wt% asphaltene pose many production problems due to asphaltene precipitation[2].

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

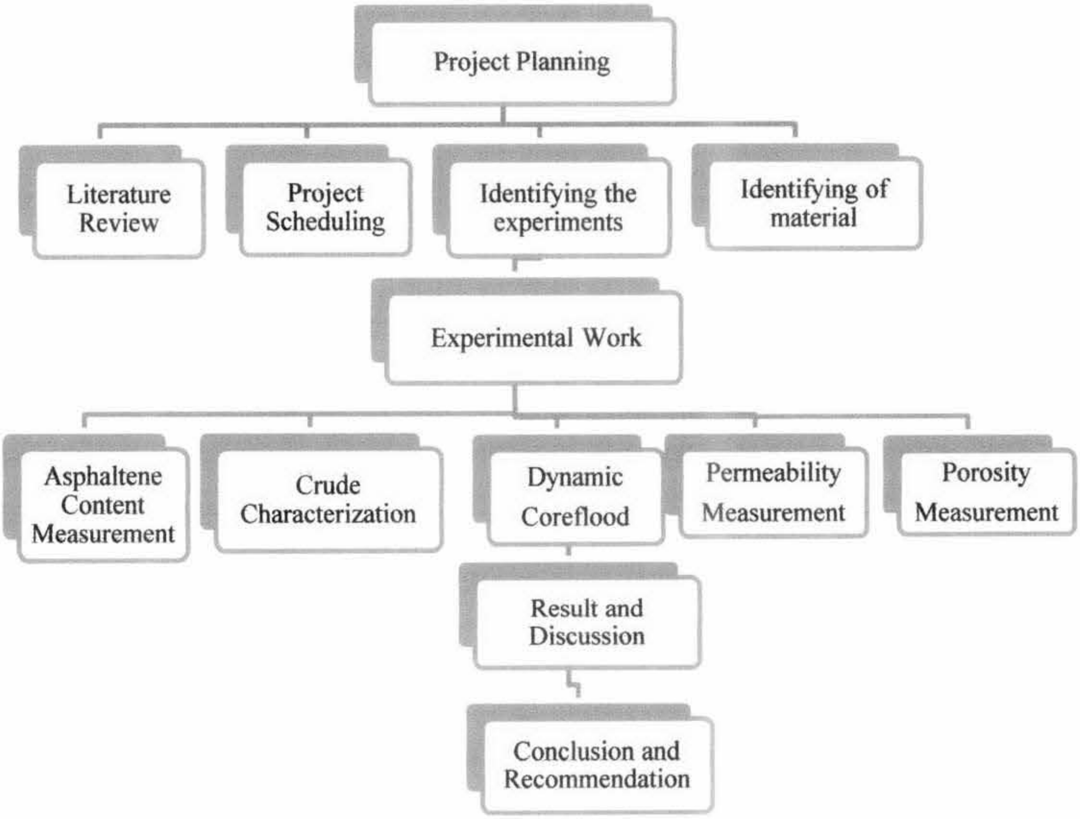

Figure 6: Research Methodology

3.2 Experimental Work

3.2.1 Crude Oil Sample

In this study, Dulang crude oil is used as an oil medium since it was a light oil sample and obtained from Dulang field which is located in the offshore Malay Basin, Malaysia. Dulang field has experienced production problems associated with asphaltene content in the crude oil. General characteristics of Dulang crude oil extracted form literatures are presented in Table 3.1.

Sample Oil	Dulang
API Gravity	38 °API
Reservoir Temperature	98 °C
Reservoir Lithology	Sandstone
Average Permeability	200 mD
Average Porosity	30 %
Oil viscosity @ 98 °C	0.80cp
Formation Water Salinity	3000-4000 ppm

Table 1: Dulang Crude Oil and Reservoir Properties [29]

3.2.2 Density Measurement

Oil density had been measured using electronic device called densometer. Data collected are presented in Table 3.3.

Sample	Average Density (g/cm^3)	Temperature (°C)	Specific Gravity	°API
Dulang	0.8343	27.0	0.837	37.56

Table 2: Measured Oil Density

3.2.3 Porosity and Permeability Measurement

3.2.3.1 POROPERM Systems

The POROPERM instrument is a permeameter and porosimeter used to determine properties of core plug at ambient confining pressure.

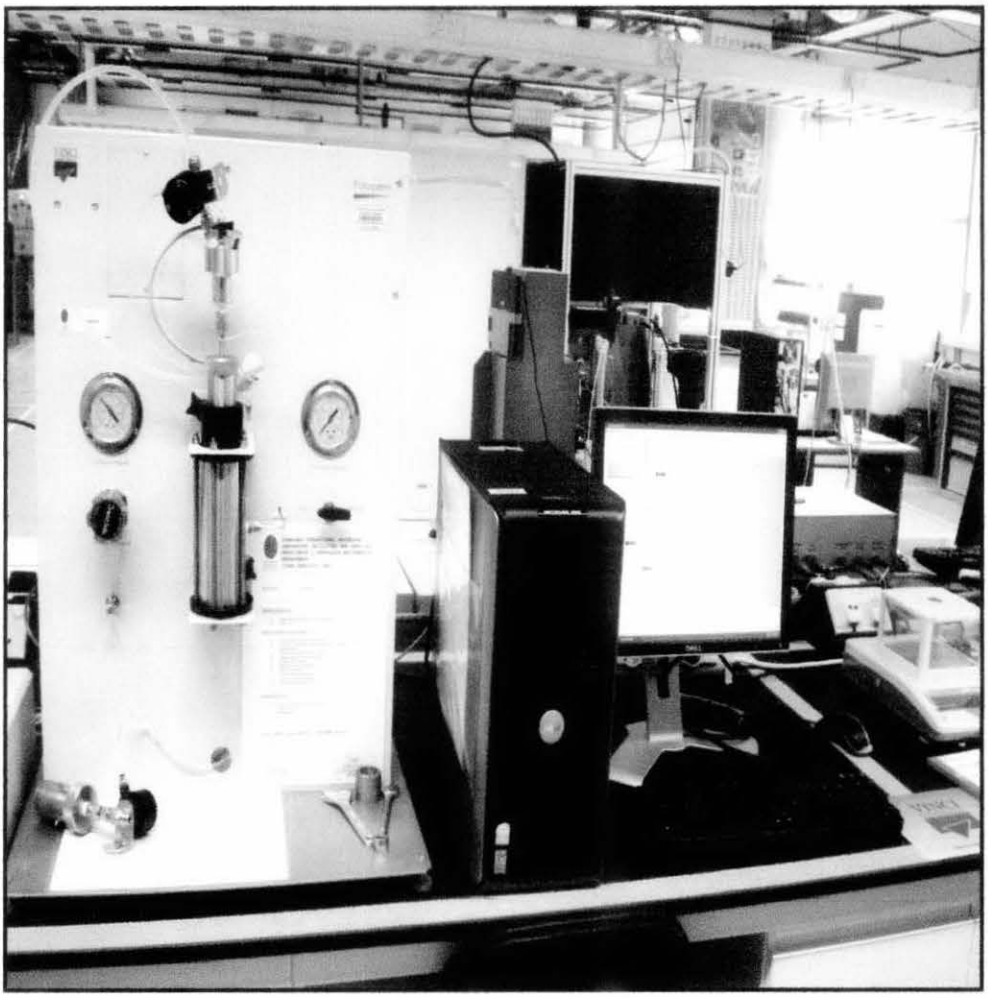

Figure 7: POROPERM Instrument

Before using PoroPerm device, a cleaned core plug was obtained and measures its weight, length, and diameter using digital caliper. The core plugs are to be put in the core holder vertically in the machine with confining pressure applied up to 200 psi. For this experiment, Helium gas was used. The system in the computer will automatically display the graphs and characteristics of the core plug.

3.2.3.2 Benchtop Permeability Systems (BPS-805)

The BPS-805 is a set of benchtop components for performing flow tests on core samples to determine specific, effective, and relative permeability. The system can be configured to use liquid and gas at ambient temperature. The standard plug size coreholder can be utilized to 10 000 psig confining pressure and up to 9 500 psig pore pressure with 1 inch and 1.5 inch diameter samples that are up to 4 inch long.

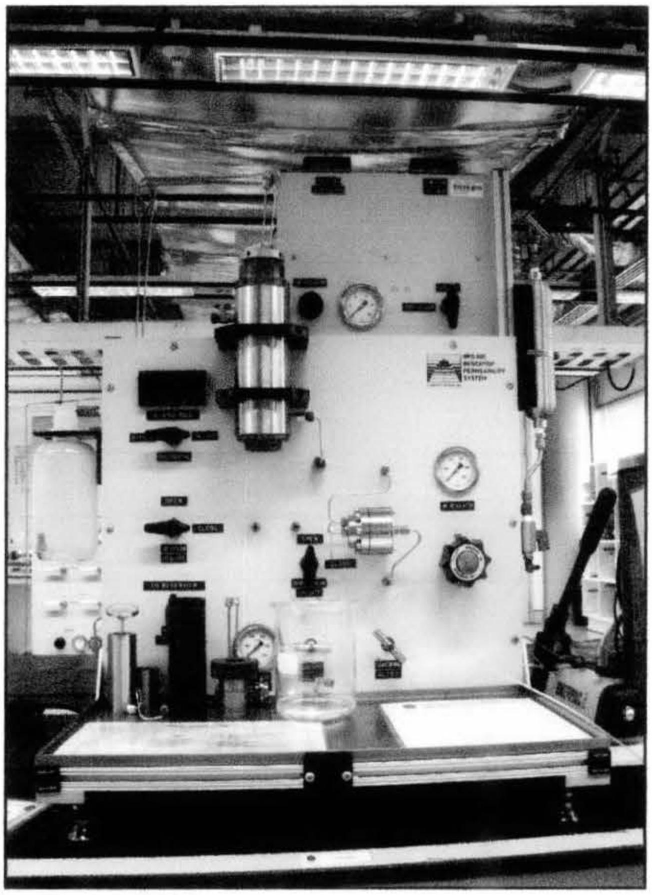

Figure 8: Benchtop Permeability Systems

There are six major components for BTS-805 which are HPLC pump for fluid delivery, Hassler type core holder, confining pressure system, differential pressure measurement system, dome loaded backpressure regulator, and optional data acquisition system consisting of BPS-805 software program.

3.2.4 Core Flooding Using Relative Permeability System (RPS)

The important part of this study is conducting core flooding test using RPS equipment. The result will determine the permeability reduction due to asphaltene precipitation during CO_2 injection.

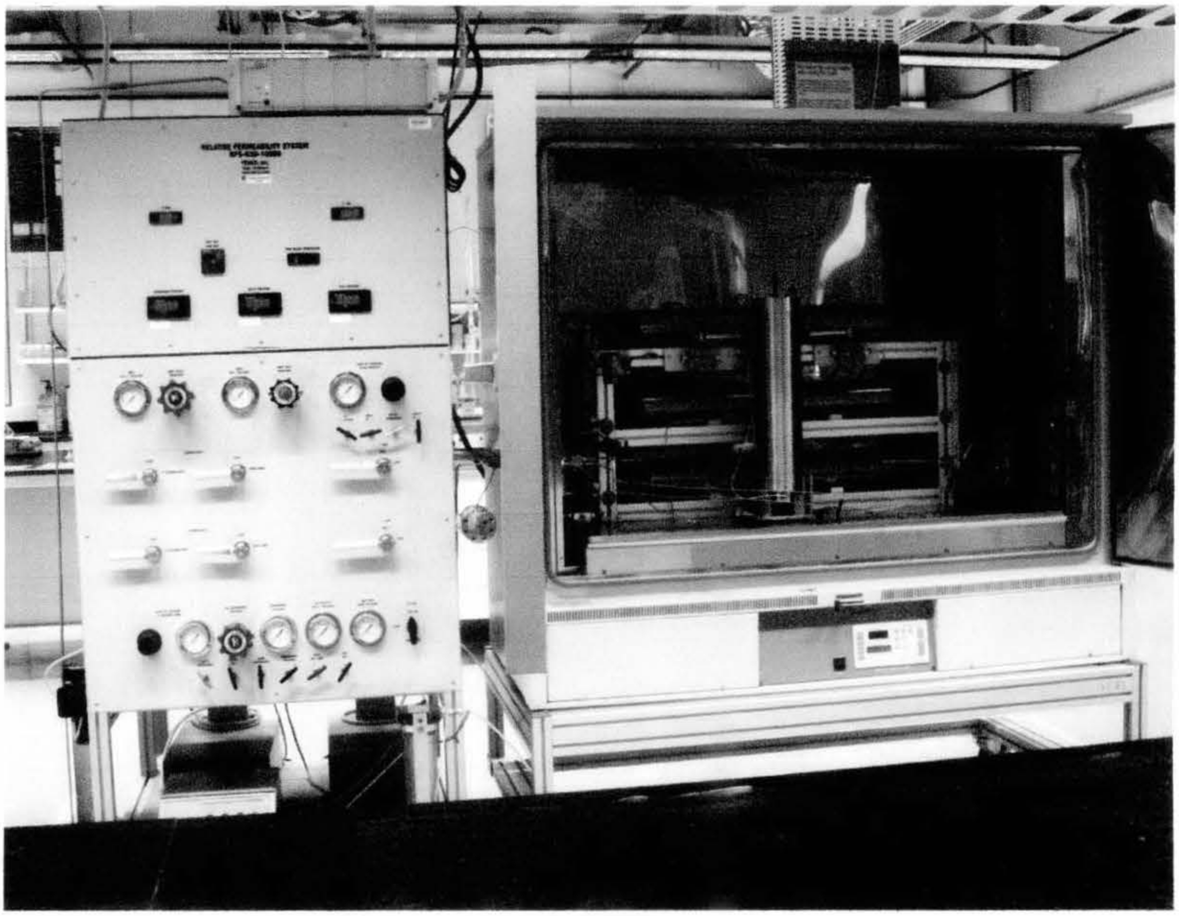

Figure 9: Relative Permeability Systems

3.2.5 Core Samples Preparation

A total of three Berea sandstone core sample with 1.5 inch diameter and 3 inch in length were used in this study. The cores were cleaned to extract all remaining fluids with Soxhlet extractor using toluene as a solvent. Then, the cores were dried in oven for 24 hours before measuring the initial porosity and permeability using PoroPerm equipment. Synthetic brine solution prepared for saturation process similar to Dulang formation water which is 4000 ppm. The cleaned and dried core was saturated in the synthetic brine under vacuum condition at least 24 hours using manual saturator.

Then, measurement of liquid permeability of saturated core with BPS will be done before conducting the core flooding.

After the core flooding, the core will be flushed with n-Heptane to clean and remove the oil inside the core. This core then dried again in the oven for 24 hours. After that, measurement of final porosity and permeability using PoroPerm machine will be done. Then, the core will be saturated again using manual saturator with pressure 1000 psi for at least 24 hours. After that, the liquid permeability of saturated core will again was measured using BPS.

3.2.6 Core Flooding Procedure

In this experiment, the core flood test will be conducted near to original reservoir condition. The objective of this project is to investigate the effect of asphaltene precipitation on absolute permeability during CO_2 injection in light oil. For this project, it was planned to conduct with three different CO_2 injection rate with constant temperature at 98°C and constant pressure at 2000 psig. During the test, the production pressure was set at reservoir pressure.

The experiment was carried out with three different CO_2 injection rates which are 0.9, 1.4 and 1.9 cc/min and injecting with synthetic brine 4000 ppm. For each run, the injection of brine and crude oil are injected at rate 1.0 cc/min. The experimental setup for the core flood tests is illustrated as in **Figure 10**.

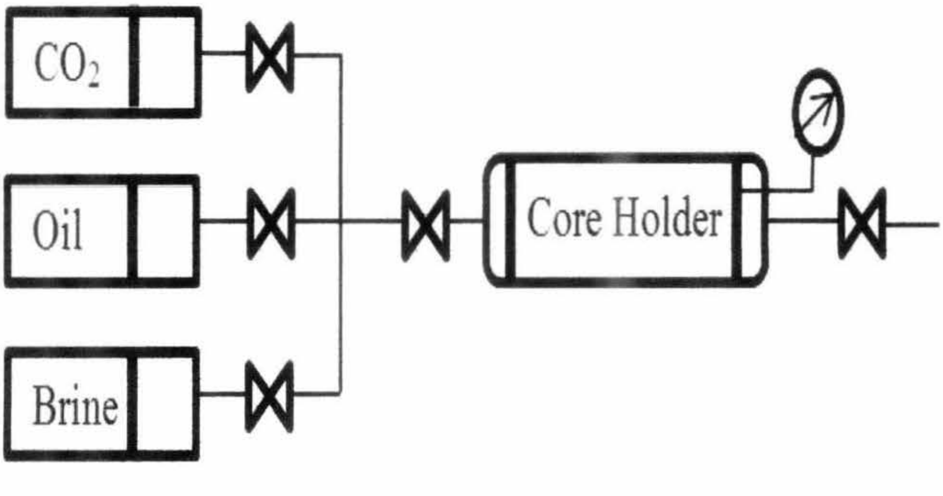

Figure 10: Schematic Diagram for RPS

First, the core sample will be injected with the brine to restore the initial condition and to determine initial permeability. After that, the injection of crude oil was started to displace the water until no more water production observed. This will determine the amount of irreducible water saturation and amount of original oil in place then calculated based on the total water volume displaced from the core at the outlet.

Then, the core was then re-injected with brine again to get secondary recovery of the reservoir oil. The amount of residual oil is calculated based on the volume of the total of produced oil. Following this process, the core sample was flooded with $CO_2$to recover remaining oil inside the core. During this stage, different rate of CO_2 injection will be used as variable parameter to determine the effect of CO_2 injection rate to asphaltene precipitation and permeability reduction. The recovered oil will be collected every 10 minutes for each run until no more oil recovered observed during the test. This recovered oil was taken for asphaltene content measurement using ASTM D3270-07 method. For this study, only first 10 minute recovered oil for each flowrate will be taken and used for asphaltene content measurement since this study only focusing to compare asphaltene precipitation at the inlet and the outlet of the core.

3.2.7 Asphaltene Content Measurement using ASTM D3279-07

For this study, asphaltene content in the crude oil sample are measured using ASTM D3279-07 method. The details of standard test will be explained in the Appendix A. This method will determine the mass percent of asphaltene which it is not soluble in n-heptane. Throughout this experiment, two important asphaltene content measurement will be determined which are initial asphaltene content before coreflooding and final asphaltene content after coreflooding.

The measurement carried out by mixing 1g of Dulang crude oil sample with 100 mL n-heptane in the Erlenmeyer flask. Then, the mixture is heated gently using magnetic stirrer hot plate and secured under reflux condenser as shown in **Figure 11**. The mixture was heated for a period of 30 minutes and then the mixture is cool down to room temperature for a period of 1 hour.

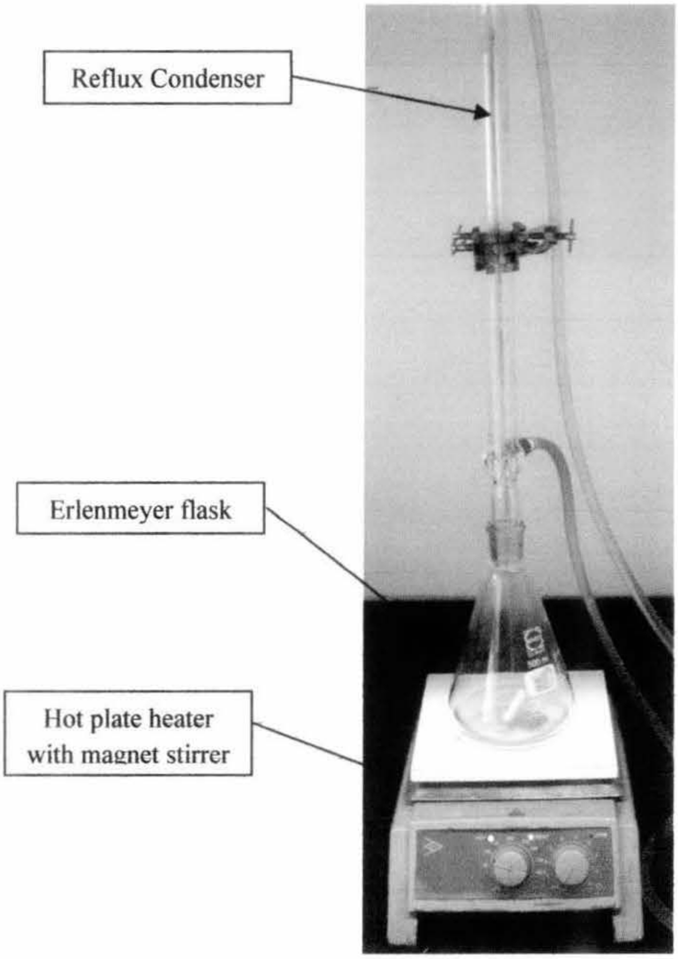

Figure 11: Experimental Setup for the Mixture Heating Process

While waiting for the mixture, a Gooch crucible with glass filter pad as **Figure 12**was heated in the oven at 107°C for 15 minutes and took into the desiccator for another 15 minutes to remove any humidity. After that, crucible and glass filter pad were weighted to obtain initial weight before filtration process.

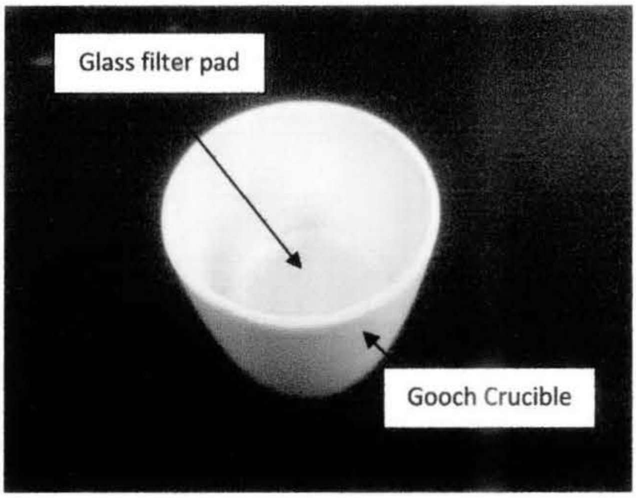

Figure 12: Gooch crucible with glass filter paper

Next, a vacuum filter is prepared with a suction flask for the filtration process as show in **Figure 13**.The flask containing the sample mixture is heated again at temperature 40°C using hot plate and poured into the crucible at the top of suction flask using a gentle vacuum. The filtration will proceed most rapidly if the supernatant liquid is filtered first with insoluble transferred to the filter last. Stainless steel spatula is used to transfer the final precipitate in the flask. The precipitate is washed with three portions of n-heptane about 10 mL each. After filtration process finish, the crucible with glass filter pad was reheated in the oven at 107°C for 15 minutes and kept in the desiccator for another 15 minutes. Then, it will weight again to get final weight after filtration process conducted.

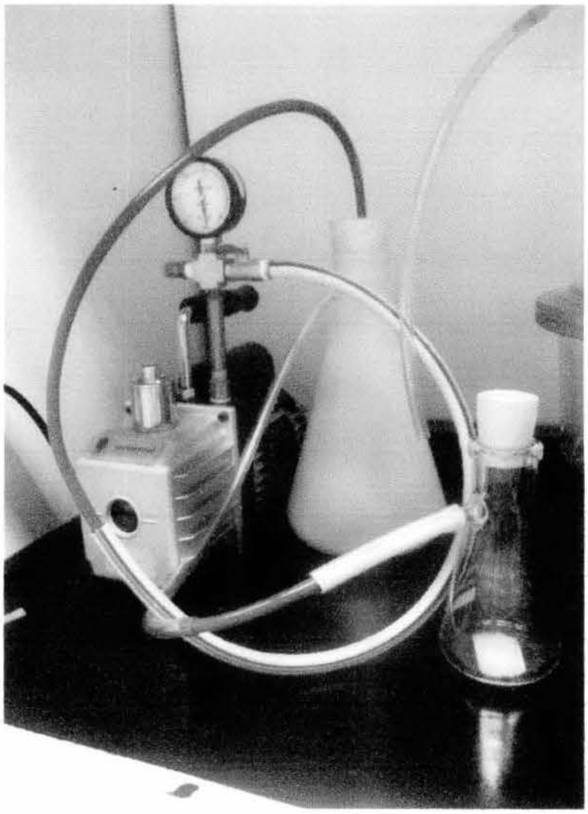

Figure 13: Filtration apparatus

As for the calculation of the mass percent of normal-heptane insolubles (NHI) as the percentage by weight of the original sample as follows:

$$\text{NHI}\ \% = (A/B) * 100 \qquad \text{...} \quad (1)$$

where:

A = Total mass of insolubles, gram

(Final weight after filtration – Initial weight after filtration)

B = Total mass of sample, gram

3.3 Project Activity and Key Milestone

	FYP 1				FYP 2			
Activities	W2 - W4	W3 - W6	W8 - W10	W11 - W13	W2 - W4	W3 - W6	W7 - W11	W12 - W14
Research on asphaltene properties and behavior	■	■						
Study on permeability and CO2 injection	■	■						
Indentify the required materials, procedures, apparatus for experiment		■	■					
Gathering and understanding the relevant method for mathematical calculation			■	■				
Conducting the experimental work					■	■	■	
Analysis and discussion of results						■	■	
Research documentation							■	■
Milestone								
Completion of asphaltene, permeability and CO2 injection mechanisms			■	■				
Completion of experimental and mathematical calculation						■	■	
Completion of analysis of results							■	
Project completion								■

Table 3: Project Activities

(FYP 2 Milestone)

No	Activities	Date/Week
1.	Progress Report Submission	W8
2.	Pre-EDX	W11
3.	EDX	W12
4.	Submission of Final Report	W12
5.	Oral Presentation	W14
6.	Submission of Hardbound Copies	W16

Table 4: Project Milestone

3.4 Gantt Chart

		FYP 1					Week								
No	**Task Name**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
1	Selection of FYP topic		■					MIDSEM BREAK							
2	Preliminary Research work			■	■										
3	Submission Preliminary Report				■										
4	Literature review				■	■	■		■	■	■	■	■		
5	Seminar					■									
6	Proposal Defense								■						
7	Draft Interim Report Submission													■	
8	Final Interim Report Submission														■

		FYP 2					Week								
No	**Task Name**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
1	Briefing of FYP 2		■					MIDSEM BREAK							
2	Update current progress			■	■										
3	Experimental Work				■	■	■		■	■	■	■	■		
4	Submission of Progress Report								■						
5	Pre-EDX											■			
6	Draft Report Submission												■		
7	Dissertation Submission (Hardbound)													■	
8	Technical Paper Submission													■	
9	Oral Presentation														■
10	Dissertation Submission (Hardbound)														■

Table 5: Gantt Chart

3.5 Tools

No	Item	Description
1	Sandstone core	Core will be used as porous media
2	Dulang crude oil	Dulang field is a light oil reservoir
3	POROPERM	To measure porosity and permeability
4	BPS	To measure porosity and permeability
5	RPS	To conduct core flooding test

Table 6: Required Tool

CHAPTER 4

RESULT AND DISCUSSION

4.1 Porosity and Permeability

4.1.1 POROPERM Measurement

Below are the result collected for the physical characterization of core samples. All measurement had been done with calibrated apparatus and equipments.

Name	C1 (0.9cc/min)	C2 (1.4cc/min)	C3 (1.9cc/min)
Diameter (mm)	37.89	37.89	37.89
Length (mm)	73.02	69.84	70.05
Initial PoroPerm System Measurement Before Coreflood			
Average Weight (g)	177.4985	169.7415	171.009
Porosity (%)	18.370	18.444	18.361
Kair (mD)	116.064	119.122	149.898
K∞ (mD)	108.764	112.19	141.539
Pore Volume (cc)	15.125	14.524	14.502
Final PoroPerm System Measurement After Coreflood			
Porosity (%)	17.803	17.640	17.526
Kair (mD)	98.437	97.808	122.978
K∞ (mD)	86.53	88.477	110.222

Table 7: POROPERM Measurement

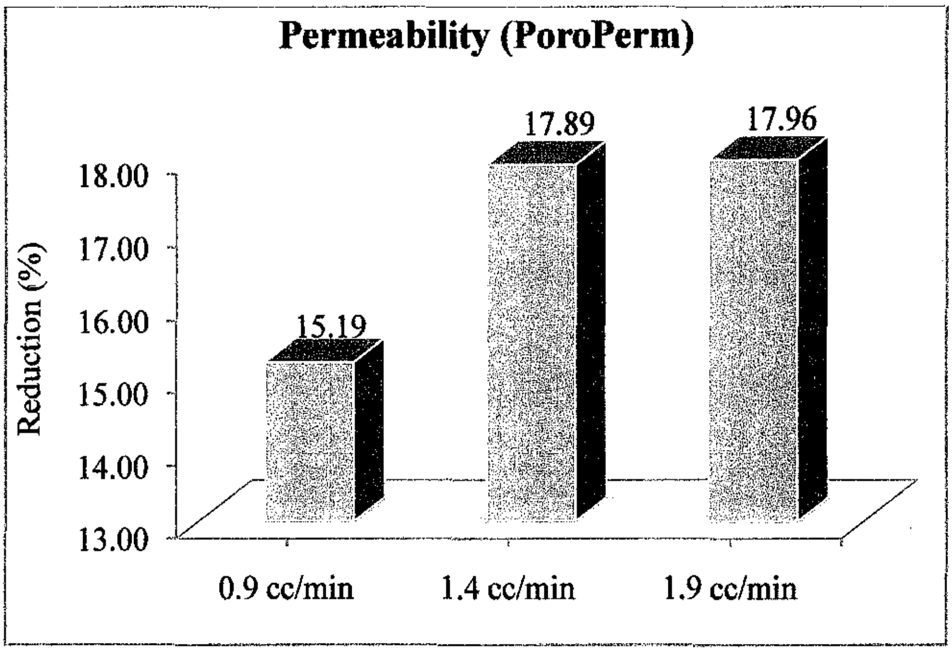

Figure 14: Bar Chart of Permeability Reduction (PoroPerm)

Based on **Figure 14** above, 0.9cc/min shows the lowest in permeability reduction which is 15.19% followed by 1.4cc/min with 17.89%. About 17.96% was the highest porosity reduction by 1.9cc/minwas recorded. The amounts of permeability reduction were obtained from the change of final to initial air permeability.

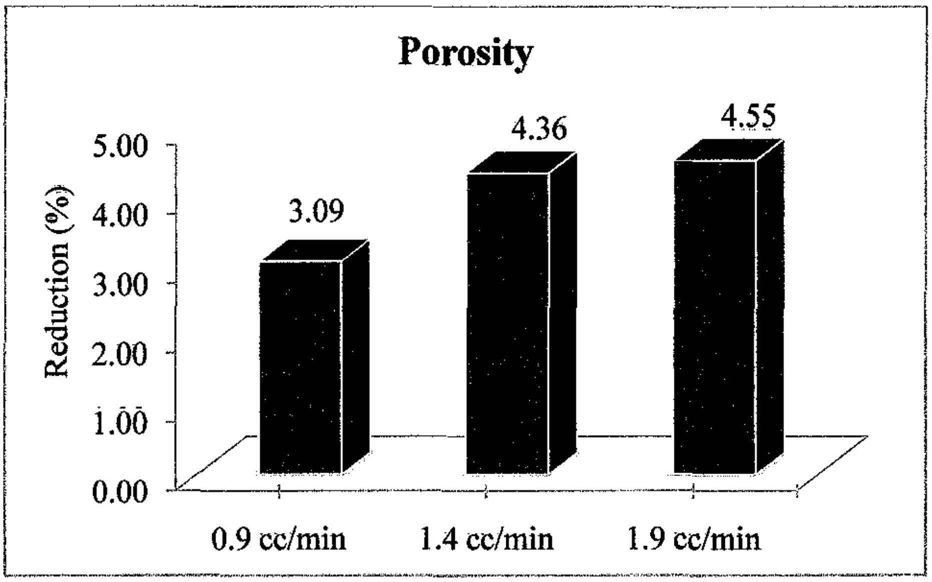

Figure 15: Bar Chart of Porosity Reduction

Same goes to porosity reduction. For **Figure 15** the C3 was the highest reduction in porosity about 4.55% while C1 was the lowest with 3.09%. About 4.36% porosity reduction was recorded for C2.The amounts of porosity reduction were obtained from the change of final to initial porosity.

4.1.2 Liquid Permeability Measurement Using BPS

Below are the result collected for liquid permeability measurement using BPS. The important parameter collected was pressure drop as stated in **Table 8.**

Name	C1	C2	C 3
Diameter (mm)	37.89	37.89	37.89
Length (mm)	73.02	69.84	70.05
Initial Stabilized Pressure Drop (psi)			
1cc/min	1.3	1.1	1.2
3cc/min	5.5	5.3	5.8
5cc/min	9.8	10.2	11.7
Average Ki(mD)	89.37	80.21	70.21
Final Stabilized Pressure Drop (psi)			
1cc/min	1.9	2.5	3.1
3cc/min	7.3	9.1	11.3
5cc/min	12.6	15.4	19.6
Average Kf (mD)	71.08	56.38	44.22

Table 8: BPS Measurement

From the measurement, **Figure 16** below shows the bar chart of liquid permeability reduction for each core with respective injections rate.

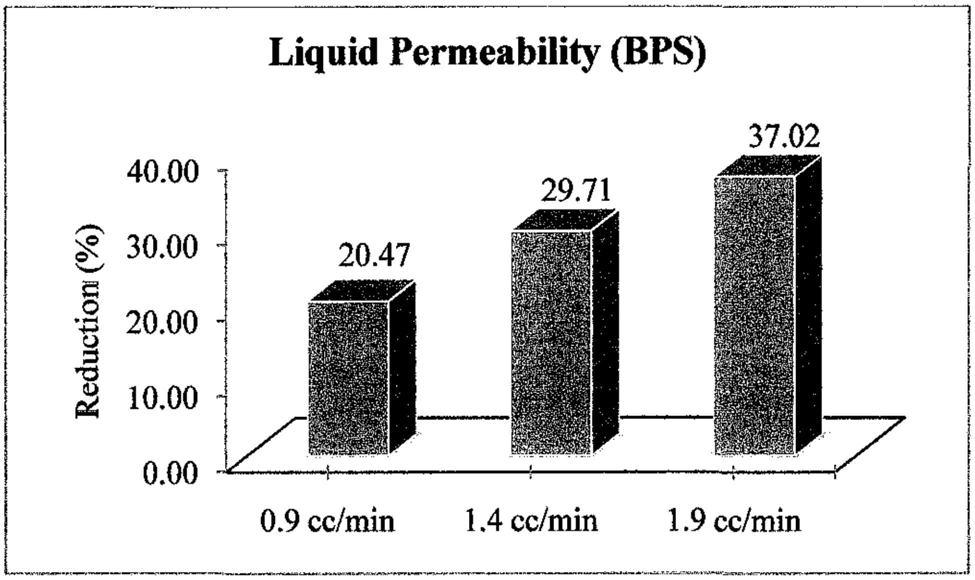

Figure 16: Bar Chart of Liquid Permeability Reduction (BPS)

As for comparison, 1.9cc/min had 37.02% of liquid permeability reduction while 1.4cc/min had 29.71% followed by 0.9cc/min with 20.47%. In order to measure this permeability reduction, thesteps involved will be discussed in the following paragraph.

The BPS computer system provides pressure drop data that will be used as input in Darcy Law to calculate initial and final liquid permeability. Below was the Darcy's Law equation:

$$Q = \frac{k\,A\,dP}{\mu\,L} \qquad \text{(2)}$$

Where: Q = flowrate, (cm^3/sec)

K = permeability (cm^2 or Darcy)

A = cross-sectional area (cm^2)

dP = pressure difference (atm)

μ= viscosity (cp)

L = core length (cm)

From the stabilized pressure drop data, the graph of flowrate versus pressure drop was plotted. In this section, only initial permeability calculation of C3 (1.9cc/min) will be discussed while details of C1 and C3 calculations will be shown in **Appendix**. Below **Figure 17**shows the graph Q versus dP for C3.

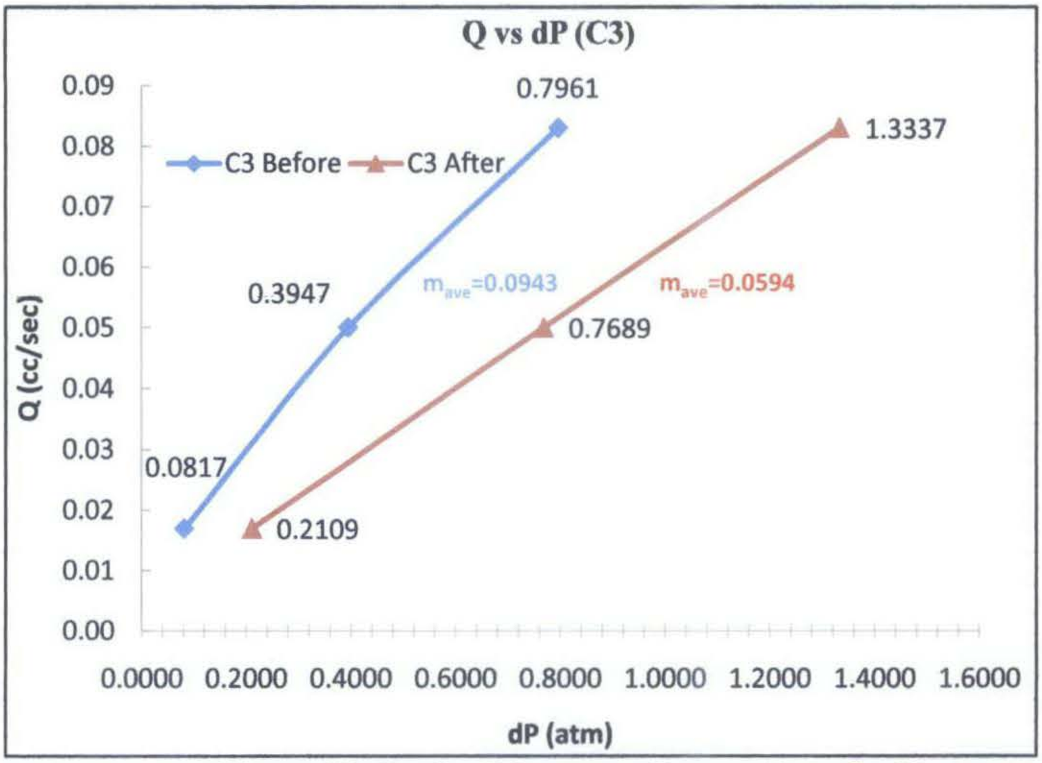

Figure 17: Graph of Flowrate vs Pressure Drop for C3

To calculate liquid permeability, below was the sample calculation for initial permeability for C3.

slope, $m = 0.0943$; $\mu = 1.2$ cp ; $A = 11.29\ \text{cm}^2$; $L = 7.005$ cm

$k = m\ \mu\ L / A$

$k = 0.0943 \times 1.2 \times 7.005 / 11.29$

$k = 0.0070207$ Darcy = 70.21 mD

From the graph, the slope was calculated. From Darcy Equation $Q = k\ A\ \Delta P / \mu\ L$ and Linear Equation $y = m\ x + c$; slope, $m = Q / \Delta P = k\ A / \mu\ L$; therefore $k = m\ \mu\ L / A$.

As a comparison for permeability reduction, **Figure 18** shows the permeability reduction comparison value for measurement between PoroPerm and BPS.

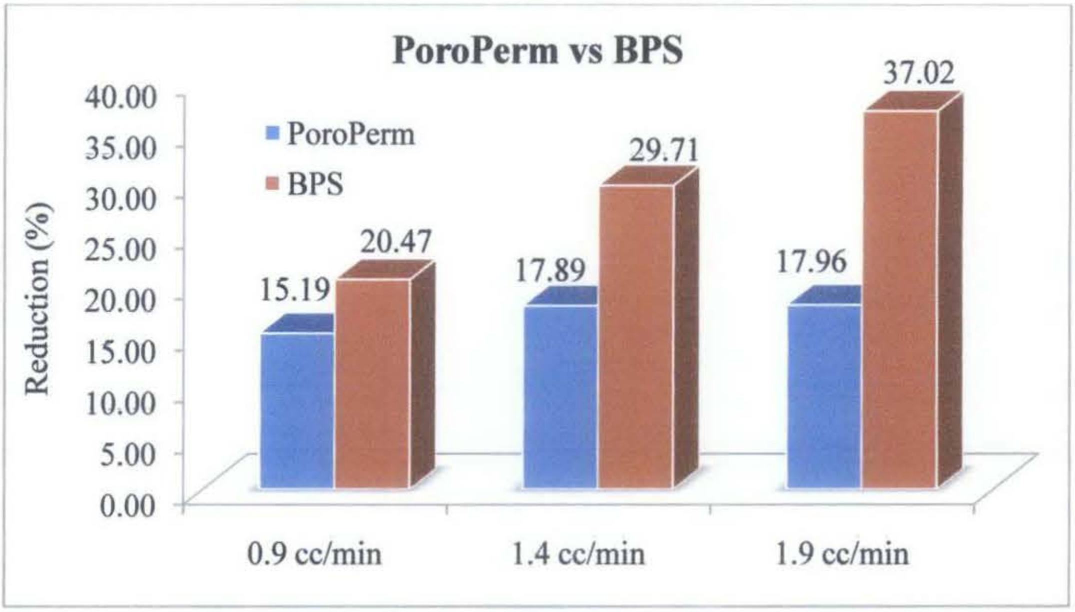

Figure 18: PoroPerm vs BPS Permeability Reduction

As show in **Figure 18**, there were some differences in liquid permeability reduction between PoroPerm and BPS measurement. For the first injection rate (0.9cc/min), BPS shows value of 20.47% reduction while PoroPerm shows of 15.19% in permeability reduction. The value for reduction using BPS start to increase when 1.4 and 1.9cc/min injections rate were used with respective value 29.71% and 37.02%. For PoroPerm results, it slightly decrease and small compare to BPS measurement. As shown at bar chart, 17.89% reduction for 1.4cc/min while 17.96% for 1.9cc/min.

For discussion, the permeability reduction in the reservoir happens due to asphaltene precipitation and deposition. The flocculated asphaltene will cause porosity reduction and permeability impairment by plugging the pore throats and wettability alterations by adsorbing on the negatively charged mineral sites (e.g., clays and silica) [2]. When asphaltene flocculation occurs in rock matrix, some asphaltene may drop out in the pores because of their larger size while the others may be carried out by the flowing fluid until they arrive simultaneously at the pore throats to bridge and reduce effective permeability.

4.3 Asphaltene Content Measurement

Theasphaltene content of Dulang crude oil before and after core flooding had been measured as shown in **Table 10** using ASTM D3279-07method.

Crude Oil Sample	Initial Asphaltene Content (wt%)	Final Asphaltene Content @ Outlet (wt%)			Asphaltene Content Left Inside Core (wt%)		
		0.9 cc/min	1.4 cc/min	1.9 cc/min	0.9 cc/min	1.4 cc/min	1.9 cc/min
Dulang	0.43	0.10922	0.10282	0.09875	0.32078	0.32718	0.33125

Table 10:Asphaltene Content Measurement

As mentioned in the **Table 10**, the initial amount of asphaltene content for Dulang crude oil was 0.43 (wt%). The oil sample was collected at the outlet in the end of the coreflood test and its final asphaltene content was measured. For 0.9cc/min injection, there was 0.10922 wt% of final asphaltene content was calculated while about 0.10282 wt% for 1.4cc/min. The lowest amount of final asphaltene content was 0.09875 wt% for 1.9cc/min injection rate. Thus the increasing of CO_2 injections rate will lead to reduction amount of final asphaltene content collected at the outlet.

In contrast, the amount of asphaltene left inside the core will be increased as the injection rate was increased. This amount of asphaltene left inside the core means the amount of precipitated asphaltene. In order to determine amount of precipitated asphaltene inside the core, the initial amount of asphaltene before coreflood will be deducted with amount of final asphaltene content measured at the outlet. In 1.9cc/min injection rate, the amount of precipitated asphaltene inside the core was 0.33125 wt% which the highest aphaltene precipitation. About 0.32718 wt% asphaltene precipitated inside the core for 1.4cc/min while for 0.9 cc/min was 0.32078 wt%. Therefore, amount of asphaltene will be precipitated directly increasing with higher CO_2 injection rate.

When injected CO_2 contacts with oil, it may be able to cause change the fluid behaviour and equilibrium which can alter the resin fraction of hydrocarbon which act as protective layer for asphaltene. This alteration gives significant effect towards

4.2 Coreflood Findings

There were three core samples (C1, C2 and C3) were used with respective (0.9, 1.4 and 1.9 cc/min) CO_2 injection rates in corefflooding. **Table 9** shows the results from coreflood test using RPS.

Core Name	C1	C2	C3
CO2 Injection Rate (cc/min)	0.9	1.4	1.9
Oil Displace Brine (ml)	14	14	13
Brine Displace Oil(ml)	7	8	8
OOIP (ml)	8.97	8.97	7.97
Oil Recovered (ml)	4.3	4.5	4.7
Oil Recovery (%) During CO_2 Injection	47.94	50.17	58.97

Table 9: Coreflood Results

For the coreflood experiment, to obtain value for OOIP, there was a correction need to be made by deducting amount of Oil Displace Brine with 5.03 ml. This 5.03 ml was the amount of liquid left behind inside the tubing.

From **Table 9** shows that as CO_2 injection increase, the amount of oil recovered also increased. Thus the sweep efficiency during CO_2 injection will increase too. This is because the sweep efficiency of CO_2 injection depends on the rate or time. More oil will be removed as the intensity of injection is increased. The rate of CO_2 breakthrough inside the core to displace the oil will also increase with increasing of injection rate. During high CO_2 injection, CO_2 gas will reduce the interfacial tension of oil surface and sweep the cumulative oil in the reservoir. As miscibility is approached, the oil phase and the CO_2 phase can flow together because of low interfacial tension and the relative increase in total volume of the combined CO_2 and oil phase compared to the water phase[3]. Unfortunately, there was some amount high molecular weight of hydrocarbon component such asphaltene will flocculated, precipitated and deposited at the pores rather than moving and mobilizing with CO_2 gas at high rate injection.

For this project, the author only focuses on permeability reduction comparison betweenat the inlet and the outlet rather than sweep efficiency. It believed that increasing in CO_2 injection rate will cause higher asphaltene precipitation at the inlet. This is because precipitation of asphaltene will reduce the effective reservoir permeability. `Bagheri et al.[30] using four different flowrate to investigate the effect on asphaltene deposition process. From the results, they had concluded that an increase in flowrate is followed by an increase in asphaltene deposition, porosity reduction and permeability impairment which is due to more pressure drop along the core. However, the mechanism of flowrate cause more pressure drop along core during gas flooding still not fully understood. Further details of precipitated asphaltene after coreflood will be discussed in the following section.

precipitation of organics solids compound such as asphaltene. On the basis of colloidal theory of asphaltene precipitation, the crude oil should be stable if the ratio of resin to asphaltene is greater than a certain value. In 1975, Koots and Speight[34] suggest the ratio of 1.25 which much smaller than 22. The deposition problem did not occur any more when the pressure fall below the bubble point pressure.Once asphaltene have been precipitated from the oil during CO_2injection, they may continue flow as suspended particles or they may deposited onto the rock surface causing plugging and formation damage[3].

From here, the CO_2 injection rates seen to provide influence on the original asphaltene content. **Table 10** clearly shows that CO_2 injection rate induces asphaltene precipitation since CO_2 can cause changes in fluid behaviour and equilibrium and also modify the ratio of asphaltene-resin. However, actual mechanism of resin is still debatable and the resin widely recognized as stabilizing agent for asphaltene. Initially at early stage, oil and CO_2 mixture has relatively low content of asphaltene during CO_2 flooding. As the injection continued, there will be an increment of asphaltene precipitation and deposition due to pressure drop along the core which caused by blockage and plugged inside the pores space. This phenomenon is probably due to the higher streaming potential associated with higher flow rate [35]. Streaming potential is one of the main factors that contribute to asphaltene precipitation.

In 1998, Ali and Islam[36] did research effect of flowrate on asphaltene deposition behaviour. They concluded that the asphaltene plugging strong dependence on flowrate with a minimal value for which steady state is reached only after initial plugging is clearly off. Most of asphaltene deposition is likely to take place the near wellbore or inlet of core injection[14]. Furthermore, asphaltene deposition rate is a function of flowrate. They explained that for a system that depends on flocculation, the deposition rate should depend on flowrate. For the case of fine deposition, alow flowrate translates into less energy for floatation, which typically means that the deposition rate would increase with increasing flowrates[36].

Compressive literature studies indicate that asphaltene deposition is due to adsorption followed by retention and plugging mechanism. **Ying et al.**[26] did a study on asphaltene deposition mechanism and its influence on development during CO_2 injection. According to their results, maximum asphaltene precipitated is around bubbling pressure with injected CO_2 increased thus amount of asphaltene precipitated also increased. Dynamic characteristic analyses of asphaltene deposition show that the higher accumulation of deposited and adsorbed asphaltene is shown mainly at the inlet of the core[26].

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusions

Coreflooding experiment was conducted on sandstone core to evaluate asphaltene precipitation in the core by injecting CO_2 gas as EOR method. From experiment, asphaltene is selectively deposited near the inlet of core during CO_2 injection that leads to permeability reduction due to large deposition at the upstream of the core. Asphaltene deposition also will be increase as consequently reduction in permeability at the core when the CO_2 injection flowrate increasing. Therefore, permeability is decreased when CO_2 injection rate increased thus cause increasing of asphaltene precipitation and deposition in light oil reservoir.

5.2 Recommendations

As a recommendation, replacing CO_2 with other injection fluids such as nitrogen (N_2) during coreflooding experiment is necessary to investigate its impact to permeability and asphaltene precipitation. Then, further understanding on asphaltene behaviour during pressure decline and gas injection is crucial to prevent asphaltene induced the permeability reduction. Using downhole core sample is highly recommended because it more representative and contain the actual amount of asphaltene. Furthermore, using different type of oil sample with different asphaltene content is also recommended for more data comparison.

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APPENDIX A

Standard Test Method for n-Heptane Insolubles

Designation: D 3279 – 07

Standard Test Method for *n*-Heptane Insolubles[1]

This standard is issued under the fixed designation D 3279; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers determination of the mass percent of asphaltenes as defined by insolubility in normal-heptane solvent. It is applicable to all solid and semi-solid petroleum asphalts containing little or no mineral matter, to gas oils, to heavy fuel oils, and to crude petroleum that has been topped to a cut-point of 343°C or higher.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* See Section 7 for a specific hazard statement.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

3. Summary of Test Method

3.1 The sample is dispersed in *n*-heptane and filtered through a glass-fiber pad. The insoluble material is washed, dried, and weighed.

4. Significance and Use

4.1 This test method is useful in quantifying the asphaltene content of petroleum asphalts, gas oils, heavy fuel oils, and crude petroleum. Asphaltene content is defined as those components not soluble in *n*-heptane.

5. Apparatus and Materials

5.1 The assembly of the dispersing apparatus is illustrated in Fig. 1 with details of the component parts as follows:

5.1.1 *Erlenmeyer Flask,* of 250-mL capacity adapted to an Allihn-type reflux condenser, each with a 35/25 ball joint.

5.1.2 *Magnetic Stirrer and Magnetic-Stirrer Hot Plate,* equipped with a voltage regulator.

5.1.3 *Gooch Crucible,* glazed inside and outside with the exception of the outside bottom surface. The approximate dimensions shall be a diameter of 44 mm at the top tapering to 36 mm at the bottom and a depth of 20–30mm.

5.1.4 *Filter Pad,* glass-fiber 32 mm in diameter.[3]

5.1.5 *Filter Flask,* heavy-wall with side tube, 500-mL capacity.

5.1.6 *Filter Tube,* 40 to 42 mm in inside diameter.

5.1.7 *Rubber Tubing,* or adapter for holding Gooch crucible on the filter tube.

NOTE 1—Other suitable assemblies permitting vacuum filtration with a Gooch crucible may be used.

6. Solvent

6.1 *n-Heptane,* 99.0 minimum mol % (Pure Grade).

7. Hazards

7.1 *n*-Heptane has a boiling point of 98°C and a flash point of −1°C, which means that it should be handled with care. It is recommended that both the reflux dispersion and filtration steps be conducted in a ventilated hood and away from flames or other sources of heat.

8. Procedure

8.1 Into the 250-mL Erlenmeyer flask, weigh to the nearest 0.1 mg a quantity of the sample to be tested, using 0.5 to 0.6 g for airblown asphalts, 0.7 to 0.8 g for asphalt paving binders and crude residues, and 1.0 to 1.3 g for gas oils and heavy fuel oils (Note 2). Add *n*-heptane in the ratio 100 mL of solvent per 1 g of sample, using proportionally less or more solvent as dependent upon the sample size. Unless the asphalt is in a granular form, heat the flask gently and turn it to cause the sample to be distributed somewhat over the bottom or lower sides of the flask.

NOTE 2—Tests show a small amount of insolubles (±0.3 mass %) remain on walls of the precipitation flask despite repeated washings. When expected level of n–C_7 insolubles is 6 % or less, use of a tared 250-mL

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[1] This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.47 on Miscellaneous Asphalt Tests.

Current edition approved Aug. 1, 2007. Published August 2007. Originally approved in 1973. Last previous edition approved in 2001 as D 3279 – 97 (2001).

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Glass filter pads No. 934-AH (Hurlbut) may be purchased from Reeve Angel and Company, Clifton, NJ.

Erlenmeyer flask is recommended. After all possible precipitate has been washed from the flask to the filtering crucible in 8.3, include the flask with the crucible for the drying, weighing, and calculation procedures in 8.3 and 9.1.

8.2 Place the Erlenmeyer flask, containing the sample plus solvent with magnetic stirrer added, on the magnetic-stirrer hot plate and secure under the reflux condenser. With the magnetic stirrer in operation, adjust for gentle refluxing for a period of 15 to 20 min when testing paving binders, fuel oils, gas oils, or crude residues. For airblown asphalts, a reflux period of 25 to 30 min is recommended. In all cases, allow the dispersed mixture to cool to room temperature for a period of 1 h.

8.3 Place the Gooch crucible plus one thickness of the glass-fiber filter pad in an oven at about 107°C for 15 min, allow to cool in a desiccator, and then weigh to the nearest 0.1 mg. Set up the filtering crucible plus filter pad in the suction flask and pre-wet with 5 mL of *n*-heptane (see Fig. 2). Warm the flask containing the sample plus solvent to 38 to 49°C on the hot plate and pour its contents (except for the magnetic stirrer) through the filter using a gentle vacuum. Filtration will proceed most rapidly if the supernatant liquid is filtered first with the insolubles transferred to the filter last. Police the beaker or flask while transferring the final precipitate, using either a rubber policeman or stainless steel spatula with a squared end. Wash the precipitate with three portions of *n*-heptane of about 10 mL each, first rinsing out the flask therewith. Place the crucible in the 107°C oven for a period of 15 min, cool in a desiccator, and weigh.

9. Calculation and Report

9.1 Calculate the mass percent of normal-heptane insolubles (NHI) as the percentage by weight of the original sample as follows:

$$\text{NHI, \%} = (A/B) \times 100 \qquad (1)$$

where:

A = total mass of insolubles, and

B = total mass of sample.

For percentages of insolubles less than 1.0, report to the nearest 0.01 %; for percentages of insolubles of 1.0 or more, report to the nearest 0.1 %.

10. Precision and Bias

10.1 Precision of the method has been determined as follows:

	Standard Deviation[A]	Acceptable Range of Two Results[A]
Single-operator	0.53 % NHI	1.51 % NHI
Multilaboratory	0.93 % NHI	2.78 % NHI

[A] These numbers represent, respectively, the (1S) and (D2S) limits as described in Practice C 670. The precision is for samples covering a range from 4.0 to 29.0 % HI.

11. Keywords

11.1 asphaltenes; heptane insolubles

APPENDIX B

Liquid Permeability Calculation

Core Name	Average Final Weight (g)	Final Pore Volume (cc)
C1 (0.9cc/min)	178.054	14.657
C2 (1.4cc/min)	169.516	13.698
C3 (1.9cc/min)	170.7655	13.843

Table 11: Final Core Properties

Core 1

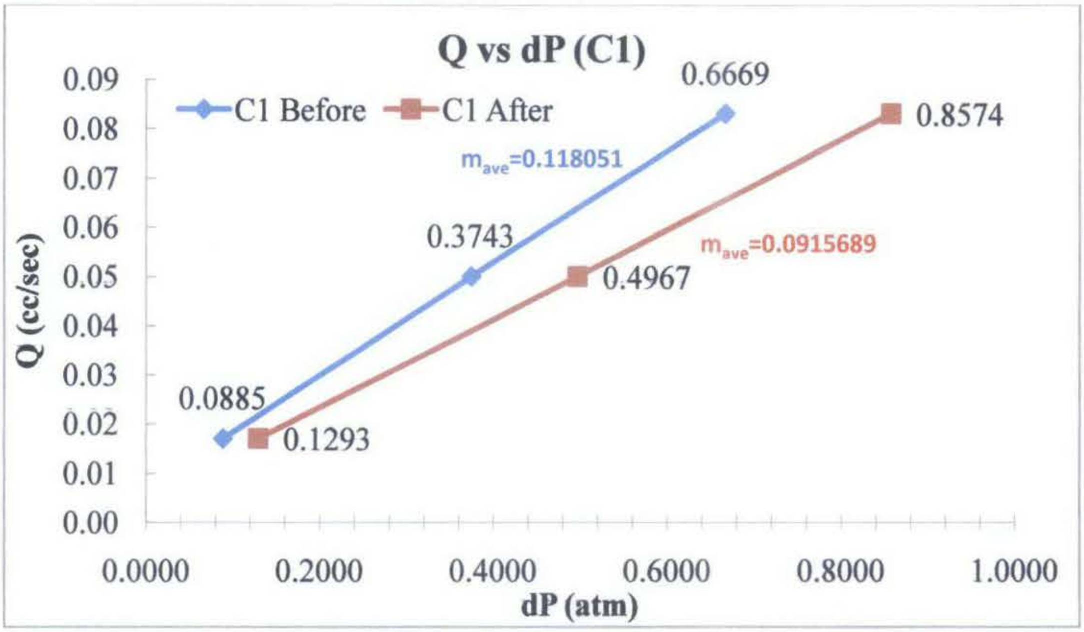

Figure 19: Graph of Flowrate vs Pressure Drop for C1

slope, m1 = 0.118051 ; μ = 1.2 cp ; A = 11.29 cm^2; L = 7.302 cm

$k = m \mu L / A$

k =0.118051 x 1.2 x 7. 302 / 11.29

k = 0.008937 Darcy = 89.37 mD

slope, m2 = 0.0915689 ; μ = 1.2 cp ; A = 11.29 cm^2; L = 7.005 cm

$k = m \mu L / A$

k =0.0915689 x 1.2 x 7. 302 / 11.29

k = 0.007108 Darcy = 71.08 mD

Core 2

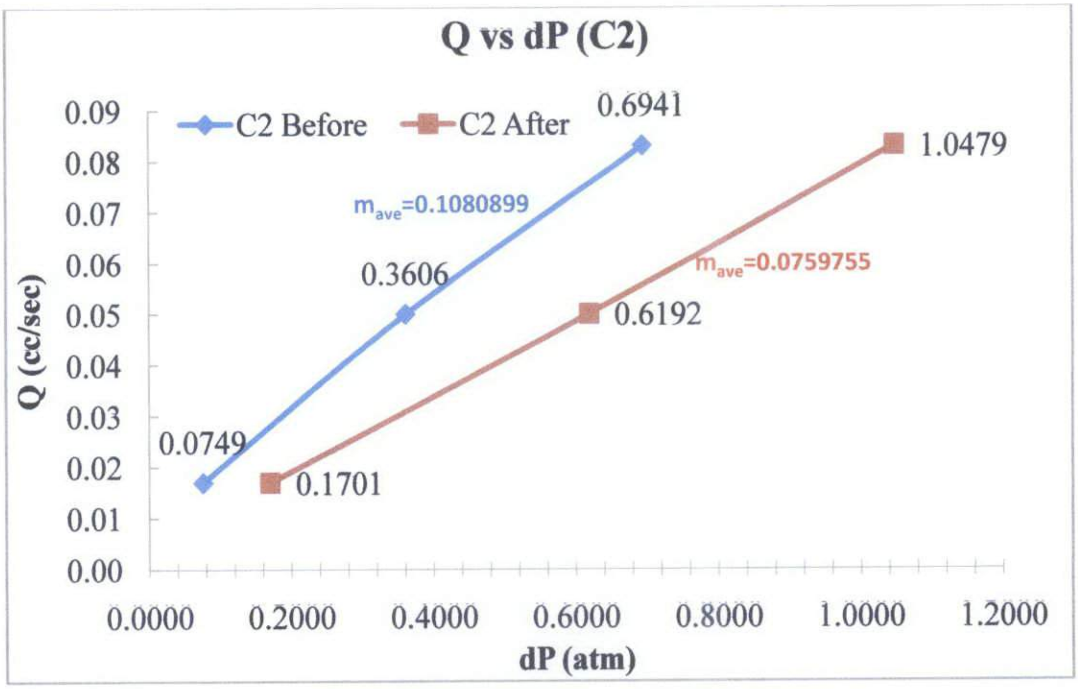

Figure 20: Graph of Flowrate vs Pressure Drop for C2

slope, m1 = 0.1080899 ; μ = 1.2 cp ; A = 11.29 cm^2; L = 6.984 cm

k = m μ L / A

k =0.1080899 x 1.2 x 6.984 / 11.29

k = 0.008021 Darcy = 80.21 mD

slope, m2 = 0.0759755 ; μ = 1.2 cp ; A = 11.29 cm^2; L = 6.984 cm

k = m μ L / A

k =0.0759755 x 1.2 x 6.984 / 11.29

k = 0.005638 Darcy = 56.38 mD