Study on Dynamic Adsorption for $BaSO_4$ Inorganic Scaling

by

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Final Report submitted in partial fulfilment of
the requirement for the
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CERTIFICATION OF APPROVAL

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A FYP Final Report submitted to the
Petroleum Engineering Programme
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TRONOH, PERAK
May 2011

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.

NOORADIAN BTE MOHD SHATAR

ABSTRACT

The technology to prevent the deposition of barium sulphate is still becoming a major challenge nowadays despite the modern equipment applied in the petroleum industry. Barium sulphate is a common downhole scale type in oil and gas wells with sea water that can reduce wells productivity. Scale inhibitor (SI) chemical treatment is the most common technique which has been applied successfully in oilfields over many years in order to prevent the formation of mineral scale. However, guidelines have been put in place to restrict the use and discharge of potentially toxic chemicals into the sea.

The main objectives of this project are to analyze the two common scale inhibitors i.e. phosphonate, DTPMPA and polyaspartate, PASP and to study PASP competitiveness as a green scale inhibitor by observing samples morphology using Scanning Electron Microscopy, SEM and its permeability using Benchtop Permeability System, BPS. Several papers were referred, namely SPE 87453 and SPE 29002 as well as from Journal of Petroleum Science 6 and Engineering by Amer Badr Bin Merdhah et al[7] (2010). Based on experiments conducted, the two SIs are compared and discussed.

ACKNOWLEDGEMENTS

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Chapter 1: Introduction

1.1. Background

In this final project report, the study of the permeability and core morphology will be discussed in the methodology and results particularly. The permeability determines the ability of a scale inhibitor to inhibit the formation of $BaSO_4$ which may lead to plugging while the brine is flowing through the core.

Currently, there are a lot of studies done to inhibit scale formation and $BaSO_4$ scale has became the topic of interest. This Final Report is dealing with an investigation on the inhibiting efficiency of two inhibitors which are polyaspartate, PASP, green scale inhibitor, GSI and phosphonate, DTPMPA, conventional scale inhibitor, CSI. This report will discuss and present the experimental results that apply the basic concept of permeability based on Darcy's equation to observe the scale inhibiting performance.

A number of theoretical and experimental studies have been conducted to investigate the mechanisms of barite formation. K. S. Sorbie et al[7] (2004) studied the two mechanisms of scale inhibitor which are nucleation inhibition and growth retardation.

1.2. Problem Statement

The oil industry is currently facing severe restrictions concerning the discharge of oilfield chemicals into the environment. International environmental statutes and guidelines have been put in place to restrict the use and discharge of potentially toxic chemicals into the sea. Since then, the objective has been put to develop environmentally friendly, biodegradable, low toxic "green" scale inhibitors (SIs) to replace the current "red" or "black" categorized conventional scale.

This project will be focusing on evaluating PASP as a GSI to replace DTPMPA, conventional SI using dynamic adsorption experimental approach.

1.3. Project Objectives

- To evaluate the ability of PASP and DTPMPA in preventing $BaSO_4$ scale deposition using dynamic adsorption experimental approach.
- To compare performance of PASP with conventional scale inhibitor, DTPMPA

1.4. Experiment objectives

- To study the permeability of core samples using poroperm meter and benchtop permeability system, BPS.
- To observe core samples morphology after each run using scanning electron microscope, SEM.

1.5. Scope of study

- Conducting research on the theory and mechanisms related to the study
- Covering the test of two scale inhibitors and observe its effect in preventing the barite scale formation as to avoid production problems at different conditions (literature review)
- As for the project working, it would be in the form of laboratory experiments and analysis

1.6. Project Relevancy and Feasibility

This study will produce a quantitative correlation and an idea of the two SIs inhibiting performance at the same pH, temperature and inhibitor concentration.

The study is expected to be feasible after much deliberation as below:

- Equipments and apparatus are available in UTP laboratory
    i. Benchtop permeability system, BPS
    ii. Poroperm Machine
    iii. Scanning Electron Microscopic (SEM)
    iv. Soxhlet extractor
    v. Desiccator and Vacuum Pump

- Scale inhibitors, DTPMPA and PASP are both readily available in UTP

- Barium chloride and sodium sulfate can be purchased in Ipoh area

Chapter 2: Theory / Literature Review

The whole study is aimed to prevent the formation of $BaSO_4$ using chemical treatment injection which is scale inhibitor. Before dynamic adsorption test is conducted, Jar Test and Tube Blocking Test are performed in order to observe whether that inhibitor exhibits competitive inhibiting performance. Scale inhibitors that will be used during this project research are phosphonates, DTPMPA and polyaspartate, PASP.

2.1 Jar Test (Scale inhibitor concentration)

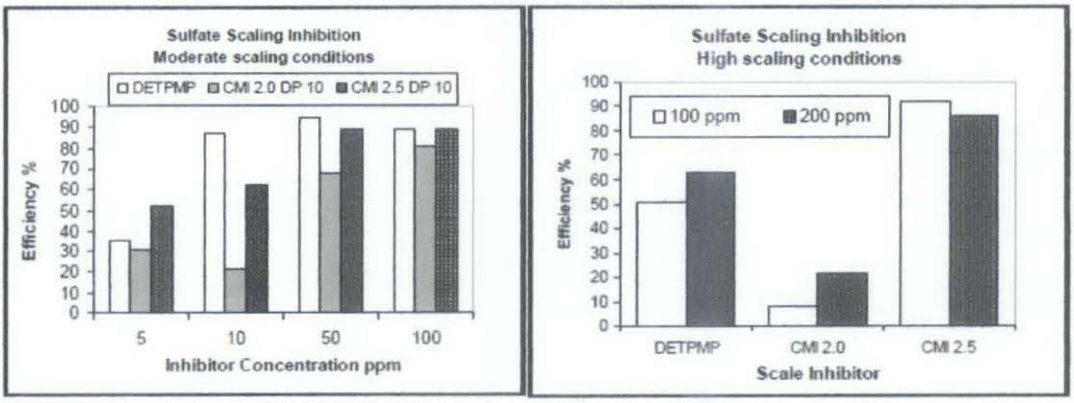


Figure 2.1: Jar Test results at different scaling conditions

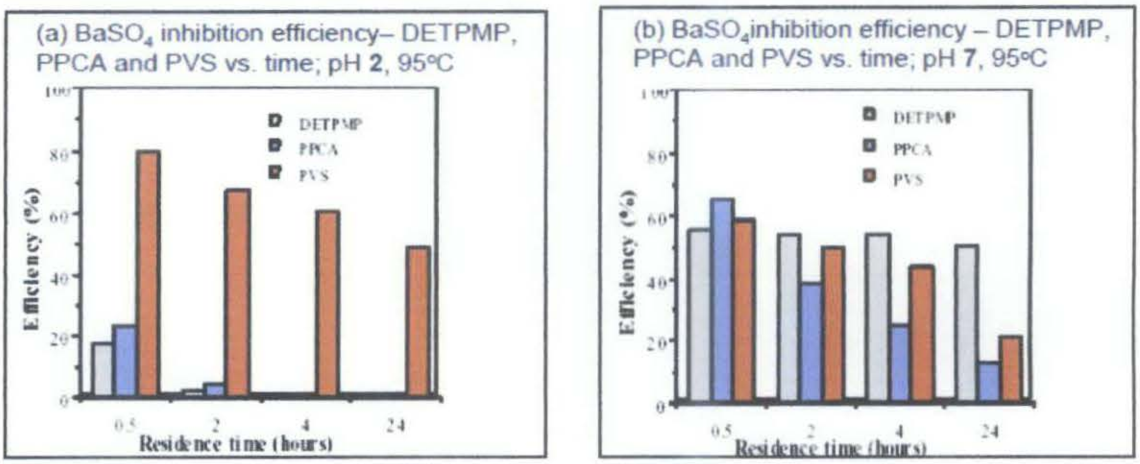
Figure 2.1 shows a Jar Test conducted and analyzed by N. Kohler et al[4] (2004). The tests were applied at different concentrations and different temperature values using the same pH value of 6.0. All the three inhibitors require high inhibitor concentration to reach sufficient $BaSO_4$ inhibiting efficiency level.

For example, in the moderate scaling condition (70°C and pH6), $BaSO_4$ scale inhibition (based on the remaining soluble Ba^{2+}) is reached by 10 to 50 ppm of DTPMPA with 85% efficiency. However, in the more severe sulphate scaling water mixture (90°C and pH6), DTPMPA fails to fully protect the water from scale deposition.

2.2 Electrostatic Repulsion Forces (pH effect)

According to H Raaijmakers et al[4] (2004), at low pH value, carboxylic groups (PASP) are less negativity charged which may increase the adsorption level by reducing the electrostatic repulsion forces between the surface and the adsorbing chemicals. This result is known after the inhibitor in sea water was injected and the concentration of effluent is measured.

Referring to SPE 87470 prepared by N. Laing et al[7] (2004) (Figure 2.2), the experiments conducted show that at a lower pH value (pH<2), DTPMPA fails to inhibit barite scale which can only give a maximum efficiency of 18%.



(a) $BaSO_4$ inhibition efficiency– DETPMP, PPCA and PVS vs. time; pH 2, 95°C

(b) $BaSO_4$inhibition efficiency – DETPMP, PPCA and PVS vs. time; pH 7, 95°C

Figure 2.2: DTPMPA inhibition efficiency at different pH values

From the patent referred, United States Patent Application Publication (US 2011/0024366)[8], under four solution pH conditions tested, results show that the amount of adsorption is lowest at the pH condition close to neutral (pH=6), higher adsorption values were observed at both acidic and alkaline condition (Table 2.1).

Table 2.1: pH effect on amount of adsorption at two different time

Solution pH	Concentration (ppm) At t = 0 hour	Concentration (ppm) At t = 24 hour	Amount of Adsorption (mg/g rock)
2	2108	1650	2.325
3.5	2114	1731	1.925
6	2148	1832	1.568
9	2128	1764	1.824

2.3 Permeability reduction

Amer Badr Mohammed Bin Merdhah (2009) did mention on correlations regarding this scale type on various parameters[2]. From the core flood test, the flow rate across the core was recorded continuously and the permeability of core was calculated using Darcy's linear-flow equation. This decrease in flow rate only occurred during the experiments when supersaturated brine was flowing through the cores. This confirms that the decrease is due to precipitation of the barium sulfates in the core with the consequent reduction in its permeability and porosity.

The reduction in permeability is possibly caused by crystals blocking the pore throats that can be observed by the Scanning Electron Microscopy, SEM. Amer Badr Mohammed Bin Merdhah also pointed out that when the concentration of brine (i.e. supersaturation) is increasing, permeability loss occurs more rapidly[3].

Darcy's Law: A proportional relationship between the instantaneous discharge rate through a porous medium, the viscosity of the fluid and the pressure drop over a given distance, L, which is:

$$Q = \frac{-\kappa A}{\mu} \frac{(P_b - P_a)}{L}$$

Equation 2.1: Darcy's Equation

Where Q: production flow rate

k: permeability
μ: viscosity
L: distance
P_b: downstream pressure
P_a: upstream pressure

The damage ratio (DR) given by:

$$\mathrm{DR} = \frac{\text{rock permeability after damage, } k_d}{\text{Original rock permeability, } k_i}$$

Equation 2.2: Damage ratio

Chapter 3: Methodology

3.1 The Experiments

The purposes of doing lab tests are to investigate core (saturated with $BaCl_2$) permeability injected with brine (Na_2SO_4) with two different scale inhibitors which are PASP and DTPMPA.

3.2 Project Activities

3.2.1 Equipments & apparatus

Among the experiment apparatus used are:

1) Poroperm equipment
2) Benchtop permeability system, BPS
3) Trimming machine
4) Memmert Universal Oven
5) Scanning electron microscopy, SEM
6) Soxhlet Extractor
7) Desiccator and Vacuum pump

3.2.2 Core Sample

The Berea sandstone petroleum core used in this experiment is provided by UTP laboratory. The basic core parameters are as follow:

Table 3.1: Core parameters

Core Parameters	
Length, L	75.27mm
Diameter, D	37.91mm
Weight, w	179.338g
Porosity, ϕ	19.51%
Pore volume, V_p	16.573cc
Grain volume, V_{grain}	68.388cc
Bulk volume, V_{bulk}	84.961cc
Grain density, ρ_g	2.622g/cc
Bulk density, ρ_{bulk}	2.11g/cc

3.2.3 Synthetic Formation and Sea Water Preparation

Both synthetic formation and sea water were made up based on the calculation below:

1. Table 3.2 shows the mass concentration of barium ion and sulphate ion that are to be used for a normal $BaSO_4$ formation.

Table 3.2: Barium ion and sulphate mass concentration

Ions	**Formation water (Barium Chloride), ppm**	**Injection Seawater (Sodium Sulphate), ppm**
Sodium	5200	10200
Potassium	105	384
Magnesium	6.6	1300
Calcium	190	400
Strontium	0.5	6.9
Barium	250	0
Chloride	6560	18520
Sulfate	0	2582
Bicarbonate	1610	151

2. Below is the calculation to obtain 250ppm barium ions and 2582ppm sulphate ions mass concentration

<u>250ppm barium ion (from $BaCl_2.2H_2O$)</u>

Molecular weight of $BaCl_2.2H_2O$ = (208.233 + 36.0296) g/mol = <u>244.2756g/mol</u>

Molecular weight of Ba = <u>137.34 g/mol</u>

% of Ba in $BaCl_2.2H_2O$ = (137.34 / 244.2756) * 100% = <u>56.22338048%</u>

Concentration of $BaCl_2 .2H_2O$ to give 250ppm barium ion

= (100% / 56.2233805%) * 250mg/L = 444.6548711mg/L

= <u>0.44466g/L</u>

<u>2582ppm sulphate ion (from Na_2SO_4)</u>

Molecular weight of Na_2SO_4 = <u>142.038 g/mol</u>

Molecular weight of SO_4= (15.9994 g/mol * 4) + 32.065 g/mol = <u>96.06 g/mol</u>

% of SO_4 in Na_2SO = (96.06 / 142.038) * 100% = <u>67.6298%</u>

Concentration of Na_2SO_4 to give 2582ppm sulphate ion

= (100% / 67.6298%) * 2582mg/L = <u>3817.844222mg/L</u>

= <u>3.81784g/L</u>

3. To prepare 11L of $BaCl_2.2H_2O$ and Na_2SO_4 solutions

Table 3.3: Brine composition

Distilled water, L	**$BaCl_2.2H_2O$ to give 250ppm Ba^{2+}'g**	**Na_2SO_4 to give 2582ppm SO_4^{2-},g**
1	044466	3.81784
2	0.88932	7.63568
3	1.33398	11.45352
4	1.77864	15.27136
5	2.2233	19.0892
6	2.66796	22.90704
7	3.11262	26.72488
8	3.55728	30.54272
9	4.00194	34.36056
10	4.4466	38.1784
11	4.89126	41.99624

3.2.4 1 Molar of NaOH and 3% of HCL in 250ml of Distilled Water Preparation

1 molar of NaOH in 250ml of distilled water

Molar mass = 40g/mol

Mass = molar mass*molar*volume of distilled water = 0.25L*1*40g/mol = 10g

3% of HCL from 37% of HCL in 250ml of distilled water

$C_1V_1 = C_2V_2$

(37%) V_1 = (3%) (250ml)

V_1 = 20.27ml HCL

3.2.5 Experiment Flow

1. Adjust pH at Block 20 laboratory for each 1L of $BaCl_2.2H_2O$ and Na_2SO_4 solutions (pH: 7) (Figure 3.1) using 1 Molar of NaOH and 3% of HCL (Figure 3.2)
2. Core cleaning. Core sample was cleaned using toluene in Soxhlet extractor for 24 hours and dried in a Memmert Universal Oven at 100°C overnight before use (Figure 3.3)
3. Measure porosity and permeability using Poroperm Equipment to determine properties of plugged samples at ambient confining pressure before it is run using BPS (Figure 3.4)
4. Saturate core with $BaCl_2.2H_2O$ for minimum of 6 hours using Desiccator and Vacuum Pump (Figure 3.5)
5. Inject scale inhibitor (PASP/ DTPMPA) into $BaCl_2.2H_2O$ (formation water) using micropipette
6. Measure viscosity using Digital Viscometer Unit at Block 20 laboratory (Figure 3.6)
7. Measure permeability of flowing fluid using Benchtop Permeability System, BPS at pore pressures of 1,900 psi with confining pressures of 2400 psi (Figure 3.7)
8. Measure porosity and permeability using Poroperm Equipment to determine properties of plugged samples at ambient confining pressure after it is run using BPS
9. Leave the core dry in a Memmert Universal Oven at 100°C overnight (Figure 3.8)
10. Cut core using Trimming Machine at Block 15 laboratory (Figure 3.9)
11. Samples were examined using SEM to observe the precipitates morphology (Figure 3.10)

Figure 3.1: $BaCl_2.2H_2O$ and Na_2SO_4 solutions

Figure 3.2: 1 Molar of NaOH and 3% of HCL

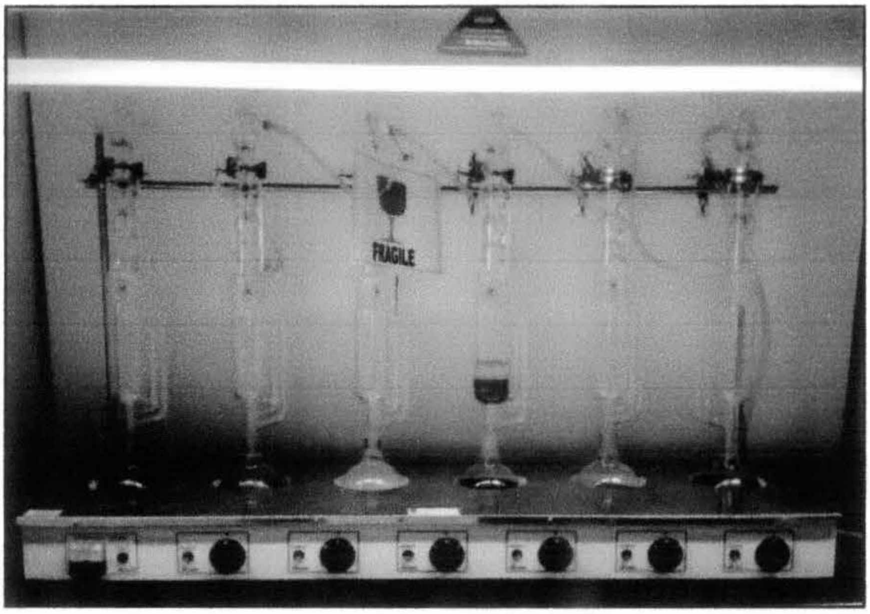


Figure 3.3: Soxhlet extractor

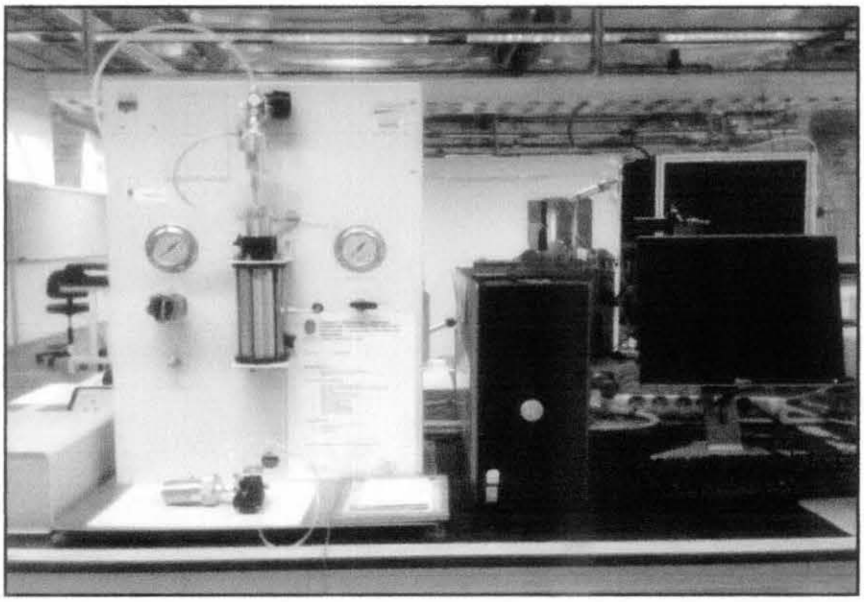


Figure 3.4: Poroperm equipment

Figure 3.5: Desiccator and Vacuum Pump

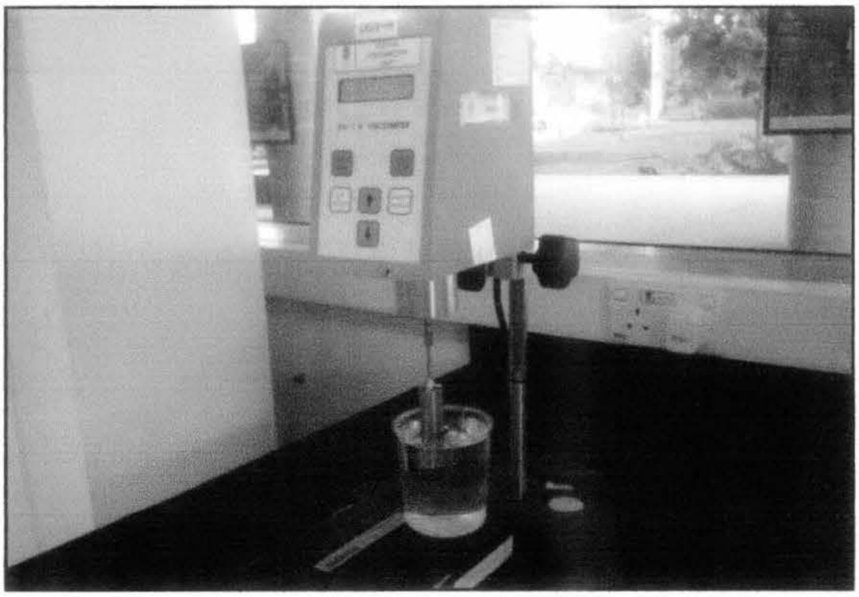


Figure 3.6: Digital Viscometer Unit

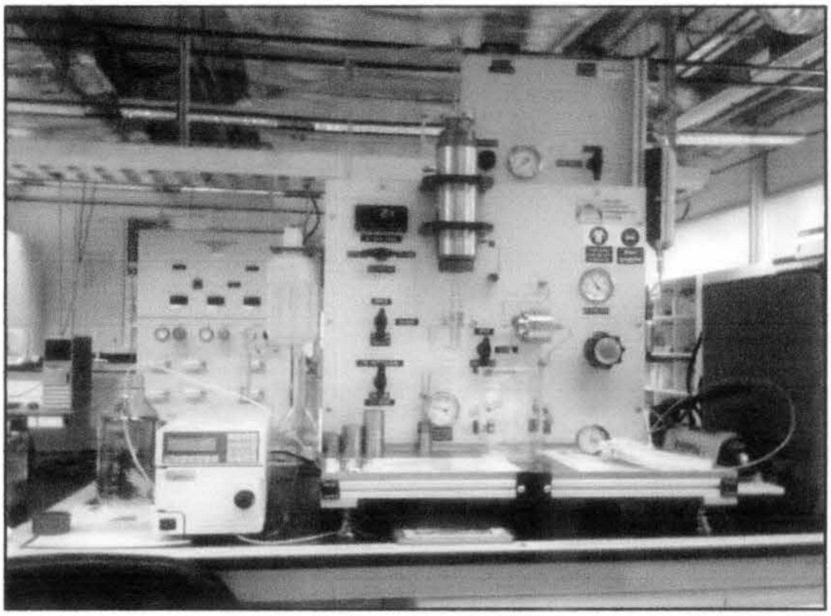


Figure 3.7: Benchtop permeability system, BPS

Figure 3.8: Memmert Universal Oven

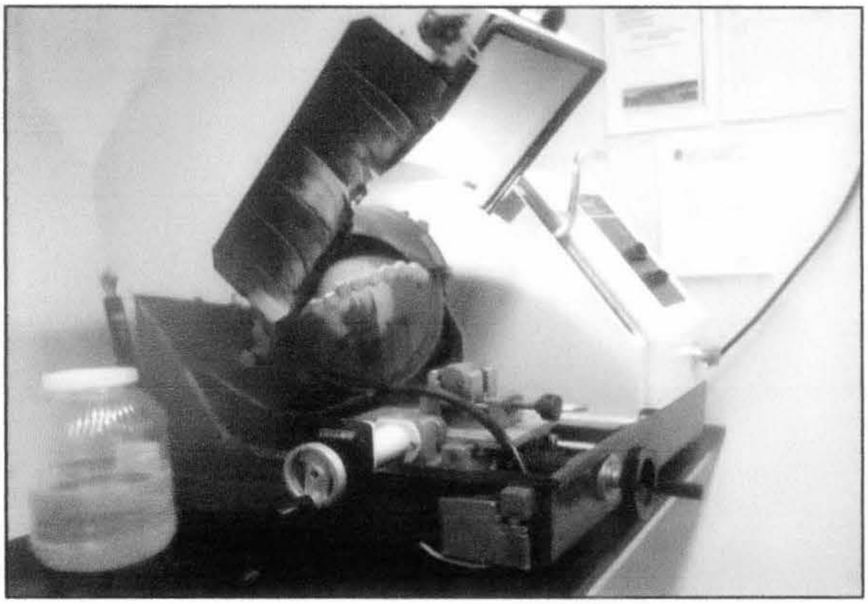


Figure 3.9: Trimming Machine

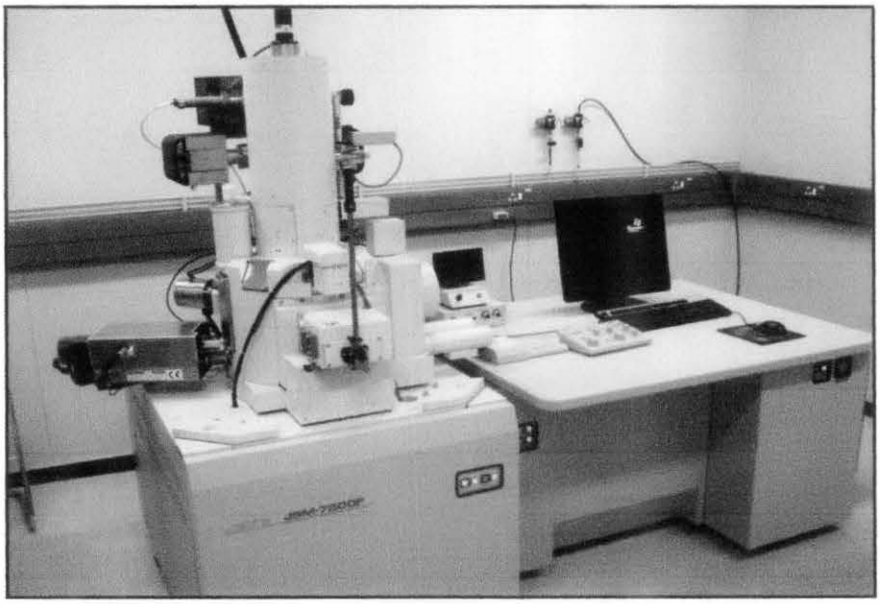


Figure 3.10: Scanning electron microscopy, SEM

3.3 Project Milestone / Gantt Chart

Table 3.4: Gantt chart

No	Detail/Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Project Work Continues	▨	▨	▨	▨	▨	▨	▨	Mid-semester break								
2	Submission of Progress Report									x							
3	Project Work Continues									▨	▨	▨	▨	▨			
4	Pre-EDX												x				
5	Submission of Draft Report													x			
6	Submission of Dissertation (Soft Bound)														x		
7	Submission of Technical Paper														x		
8	Oral Presentation															x	
9	Submission of Project Dissertation (Hard Bound)																x

x Milestone
▨ Process

Chapter 4: Results and Discussion

The main objective of the discussion part is to study the permeability reduction caused by $BaSO_4$ scale deposition in porous media with and without the presence of scale inhibitor. Due to time constraint, only 3 runs were conducted. First, with the injection of GSI which is PASP. Second, without the presence of SI. Third, with the injection of CSI which is DTPMP. Results were obtained as follow:

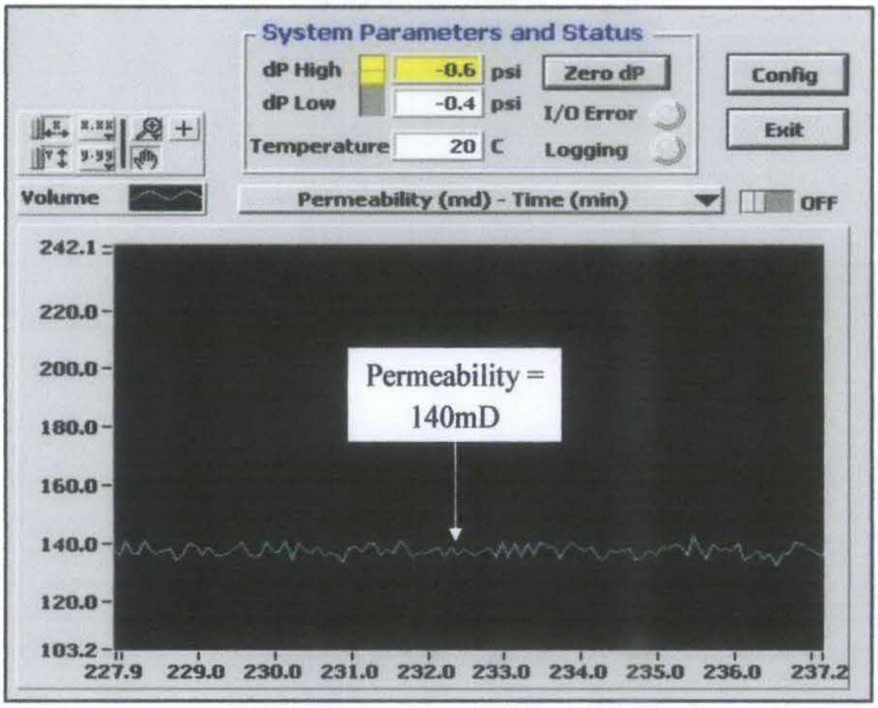
Run 1 (pH=7, SI=PASP, SI Concentration=5000ppm) results

1. Poroperm results (before BPS is run)

Parameters: L = 75.27mm
D = 37.91mm
W = 179.338g

Results: k_{air} = 132.101mD
Porosity= 19.507%
V_p=1 6.573cc
V_{grain}= 68.388cc
V_{bulk}= 84.961cc
ρ_g= 2.622g/cc
ρ_{bulk}= 2.11g/cc
P_c= 308psi

2. BPS results



System Parameters and Status

dP High -0.6 psi

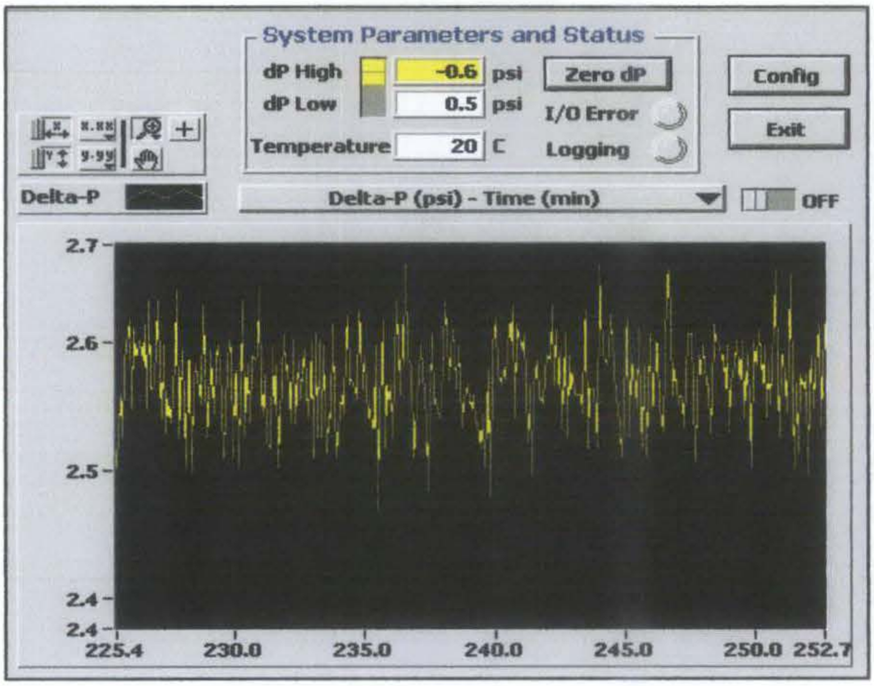
dP Low -0.4 psi

Temperature 20 C

Volume

Permeability (md) - Time (min)

Figure 4.1: Permeability (mD) vs Time (min) (Run 1)



System Parameters and Status

dP High -0.6 psi

dP Low 0.5 psi

Temperature 20 C

Delta-P

Delta-P (psi) - Time (min)

Figure 4.2: Delta P (psi) vs Time (min) (Run 1)

For the first run, conducted at pH 7, using PASP as a SI with a concentration of 5000ppm, the permeability obtained is high which is 140mD with a constant and low delta pressure (Figure 4.1 & Figure 4.2) showing less restriction across the pore throats.

3. Poroperm results (after BPS is run)

Parameters: $L = 71.34mm$
$D = 38.04mm$
$W = 167.6g$

Results: $k_{air} = 201.633mD$
Porosity= 20.588%
V_p= 16.692cc
V_{grain}= 64.386cc
V_{bulk}= 81.078cc
ρ_g= 2.603g/cc
ρ_{bulk}= 2.603g/cc
P_c= 301psi

4. Sample 1 after core trimming

Figure 4.3: Sample 1

5. SEM results that show Sample 1 core morphology

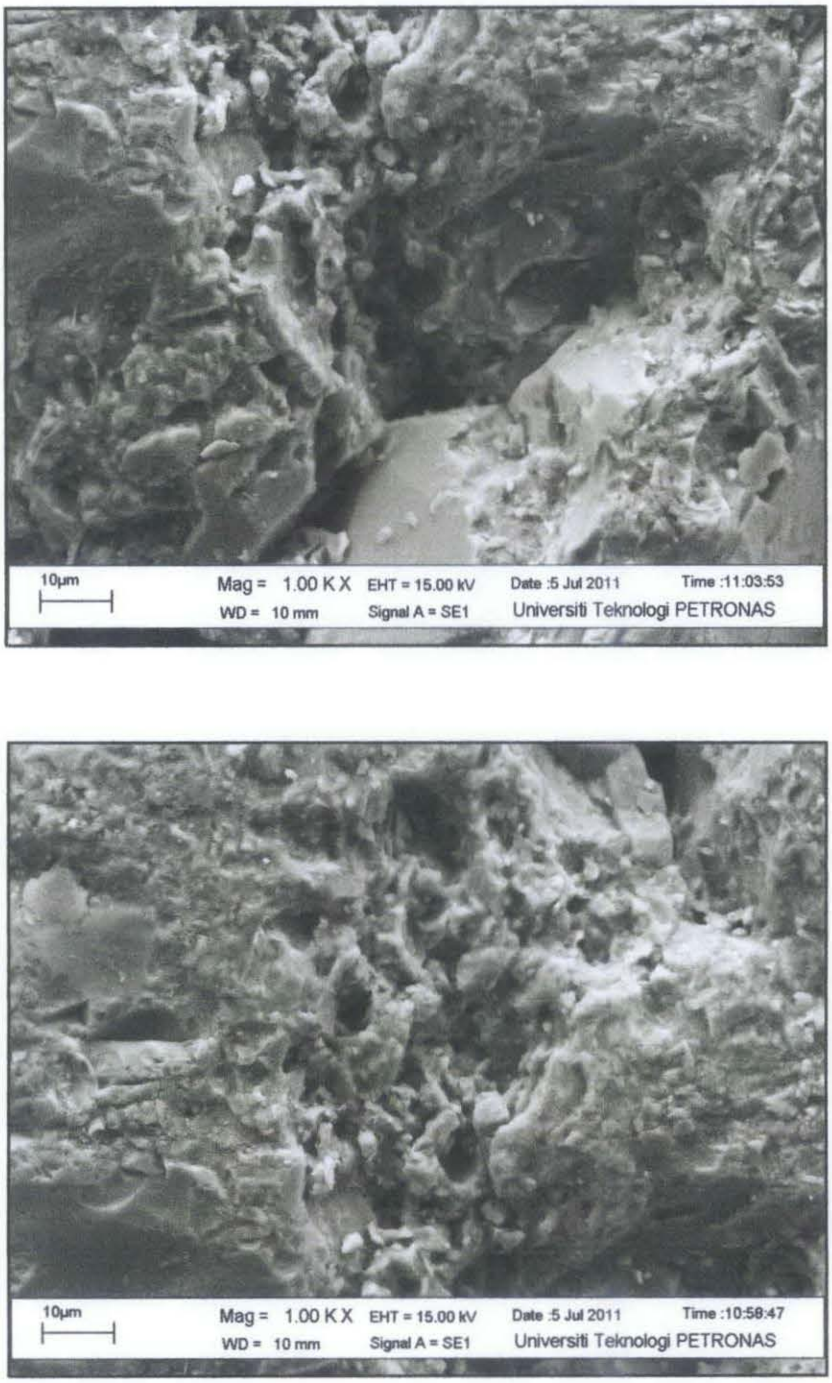


Figure 4.4: Sample 1 precipitates morphology using SEM

Figure 4.4 shows precipitates morphology where from here no crystals blocking were observed through the pore throats from the SEM images obtained. Sea water injection with the presence of PASP gave almost zero $BaSO_4$ precipitation.

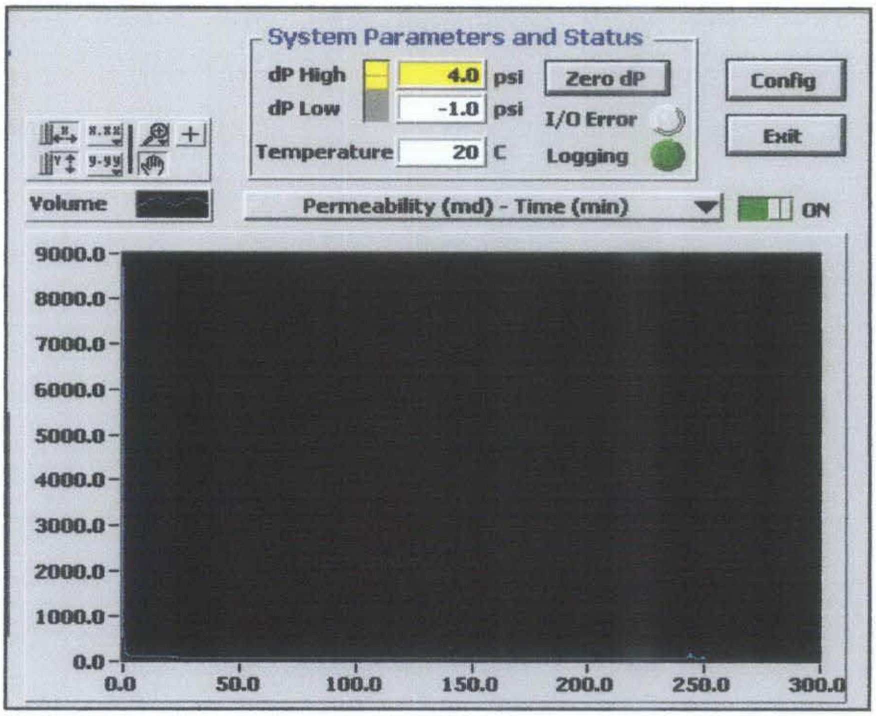
Run 2 (pH=7, without scale inhibitor injection) results

1. Poroperm results

Parameters: L = 71.5mm
D = 38.06mm
W = 167.65g

Results: k_{air} = 200.945mD
Porosity= 20.64%
V_p=1 6.789cc
V_{grain}= 64.556cc
V_{bulk}= 81.345cc
ρ_g= 2.597g/cc
ρ_{bulk}= 2.061g/cc
P_c= 304psi

2. BPS results



System Parameters and Status

dP High 4.0 psi

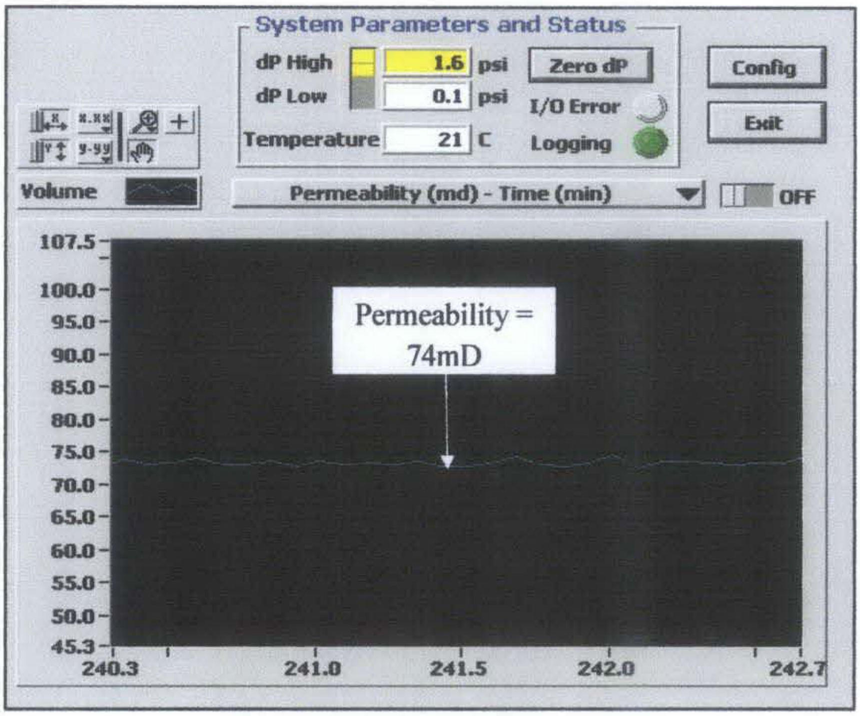
dP Low -1.0 psi

Temperature 20 C

Permeability (md) - Time (min)

Figure 4.5: Permeability (mD) vs Time (min) (Run 2)

During initial flow period, the permeability declined sharply soon after $BaSO_4$ and Na_2SO_4 are mixed in the pores (Figure 4.5).


System Parameters and Status

dP High 1.6 psi

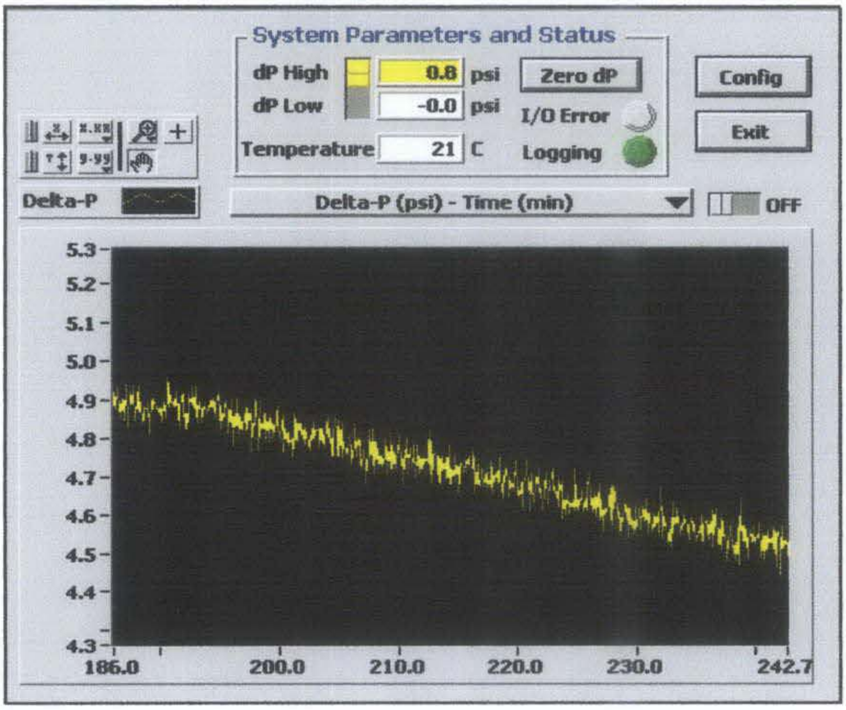
dP Low 0.1 psi

Temperature 21 C

Permeability (md) - Time (min)

Permeability = 74mD

Figure 4.6: Magnified Permeability (mD) vs Time (min) (Run 2)


System Parameters and Status

dP High 0.8 psi

dP Low -0.0 psi

Temperature 21 C

Delta-P (psi) - Time (min)

Figure 4.7: Delta P (psi) vs Time (min) (Run 2)

For the 2nd run, conducted at pH 7, without the presence of SI, the permeability obtained is very low which is around 74mD with inconsistent and quite high delta pressure showing a presence of restriction. The reduction in permeability is possibly caused by crystals blocking through the pore throats as shown in the SEM of Figure 4.9. Figure 4.7 shows that a very high pressure drop of 4.9psi leads to a very low permeability value of 74mD.

3. Poroperm results (after BPS is run)

Parameters: $L = 71.23mm$
$D = 38.10mm$
$W = 167.698g$

Results: $k_{air} = 199.789mD$
Porosity=20.487%
V_p=16.637cc
V_{grain}=64.572cc
V_{bulk}=81.209cc
ρ_g=2.597g/cc
ρ_{bulk}=2.065g/cc
Pc= 304psi

4. Sample 2 after core trimming

Figure 4.8: Sample 2

5. SEM results that show Sample 2 core morphology

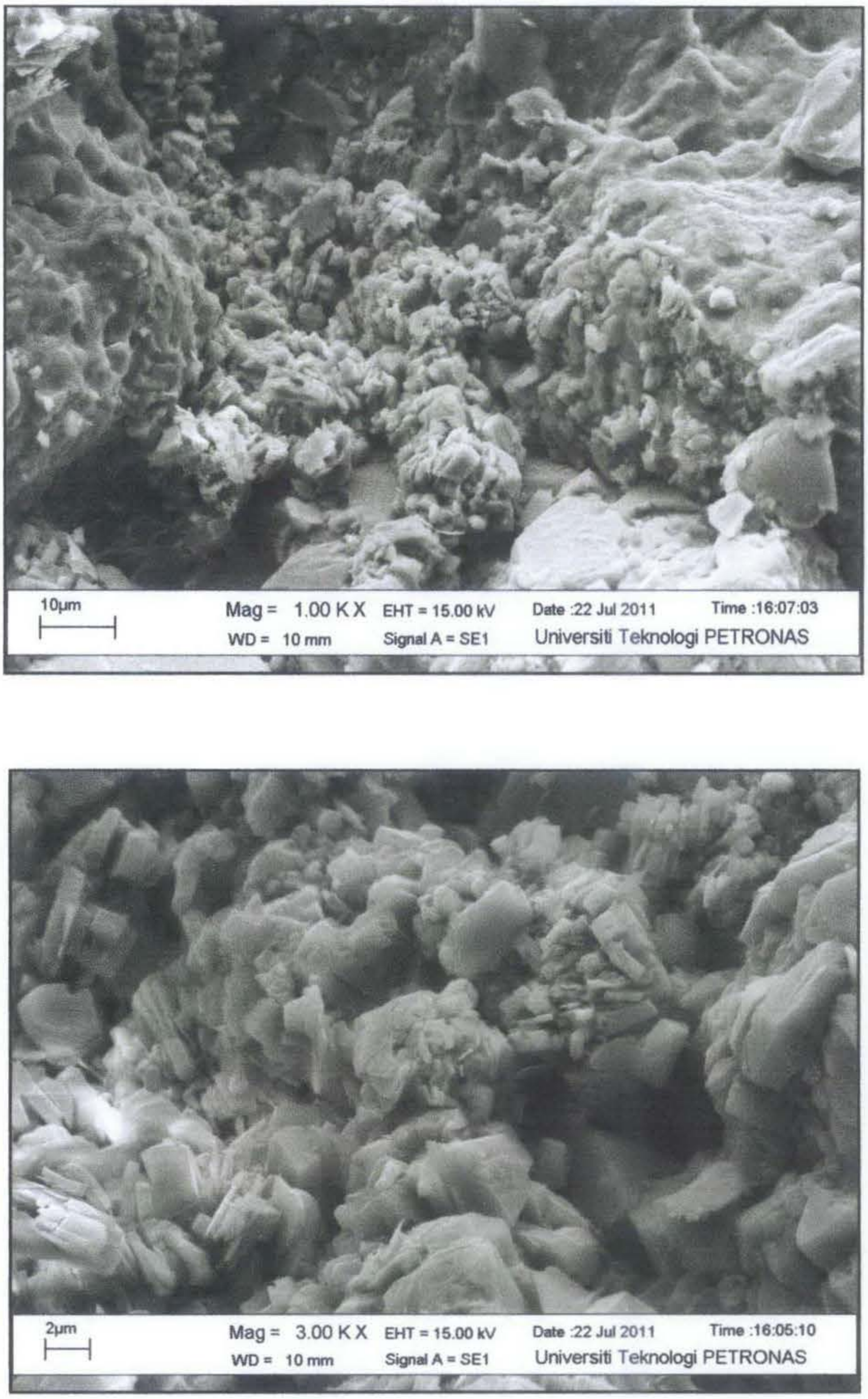


Figure 4.9: Sample 2 precipitates morphology using SEM

Figure 4.9 shows the precipitates morphology from mixed sea water and formation water inside the cores where from here we can observe crystals through the pore throats that can reduce the permeability of the fluids flow through pore throats. These crystals blocking are $BaS0_4$ that can lead to typical problems such as:

- Preventing movement of sliding sleeves
- Perforation blockage
- Blocked nipples
- Lining inside the wellbore
- Reduced formation porosity and permeability near the wellbore

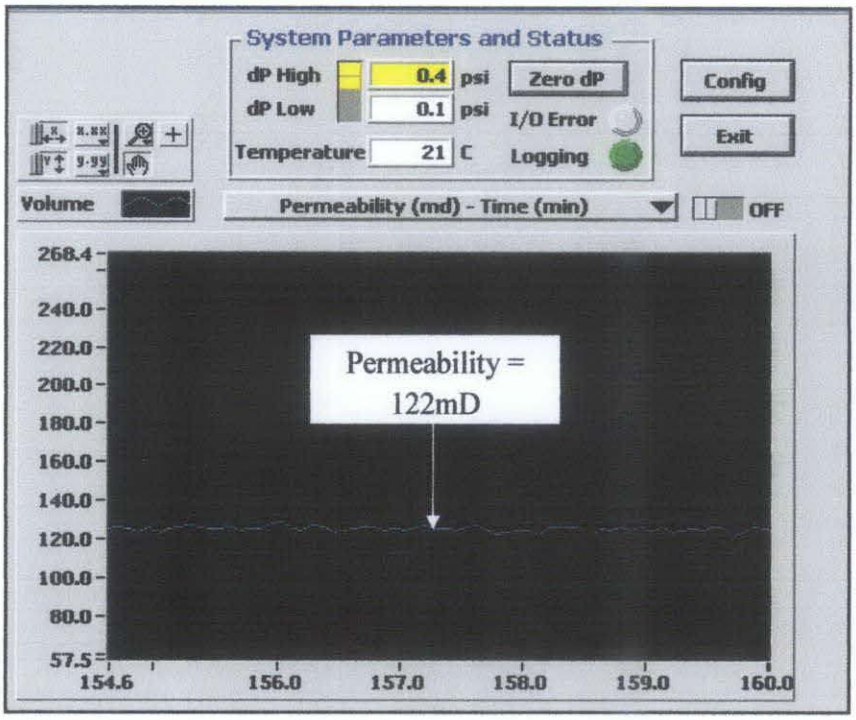
Run 3 (pH=7, SI=DTPMPA, SI Concentration=5000ppm) results

1. Poroperm results

Parameters: L = 65.97mm
D = 38.04mm
W = 155.366g

Results: k_{air} = 200.898mD
Porosity= 20.552%
V_p= 15.409cc
V_{grain}= 59.566cc
V_{bulk}= 74.975cc
ρ_g= 2.608g/cc
ρ_{bulk}= 2.072g/cc
P_c= 304psi

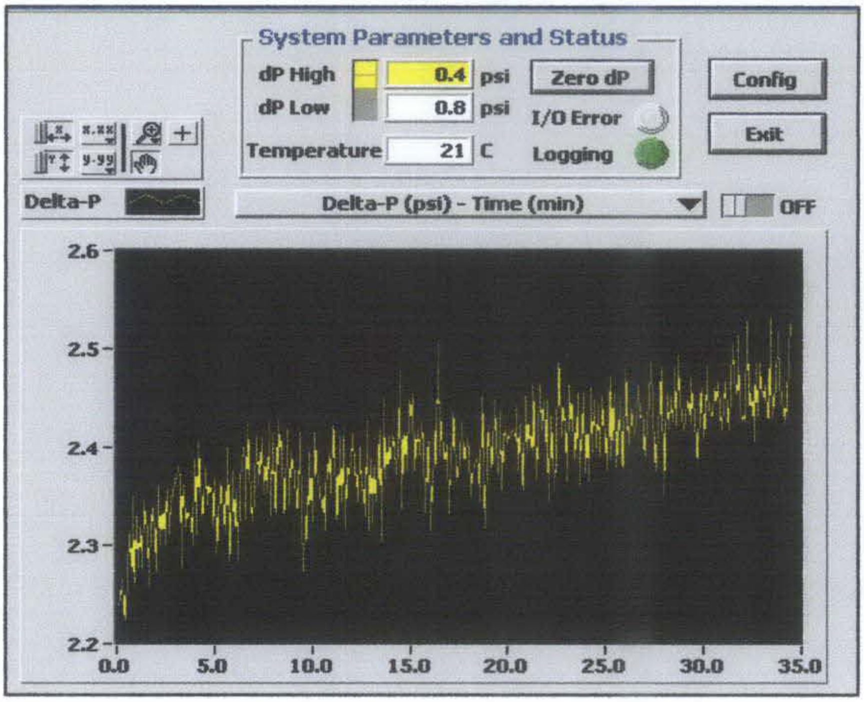
2. BPS results



dP High 0.4 psi
dP Low 0.1 psi
Temperature 21 C

Permeability =
122mD

Figure 4.10: Permeability (mD) vs Time (min) (Run 3)



dP High 0.4 psi
dP Low 0.8 psi
Temperature 21 C

Figure 4.11: Delta P (psi) vs Time (min) (Run 3)

For the 3rd run, conducted at pH 7, using DTPMPA as a SI with a concentration of 5000ppm, the permeability obtained is quite high which is 122mD with an inconsistent and quite low delta pressure showing a less presence of restriction. However, the permeability value is lower than the permeability of the first run (with PASP injection).

3. Poroperm results (after BPS is run)

Parameters: $L = 66.12mm$
$D = 38.03mm$
$W = 155.198g$

Results: $k_{air} = 186.619mD$
Porosity= 21.467%
$V_p = 16.123cc$
$V_{grain} = 58.983cc$
$V_{bulk} = 75.106cc$
$\rho_g = 2.631g/cc$
$\rho_{bulk} = 2.066g/cc$
Pc= 289psi

4. Sample 3 after core trimming

Figure 4.12: Sample 3

5. SEM results that show Sample 2 core morphology

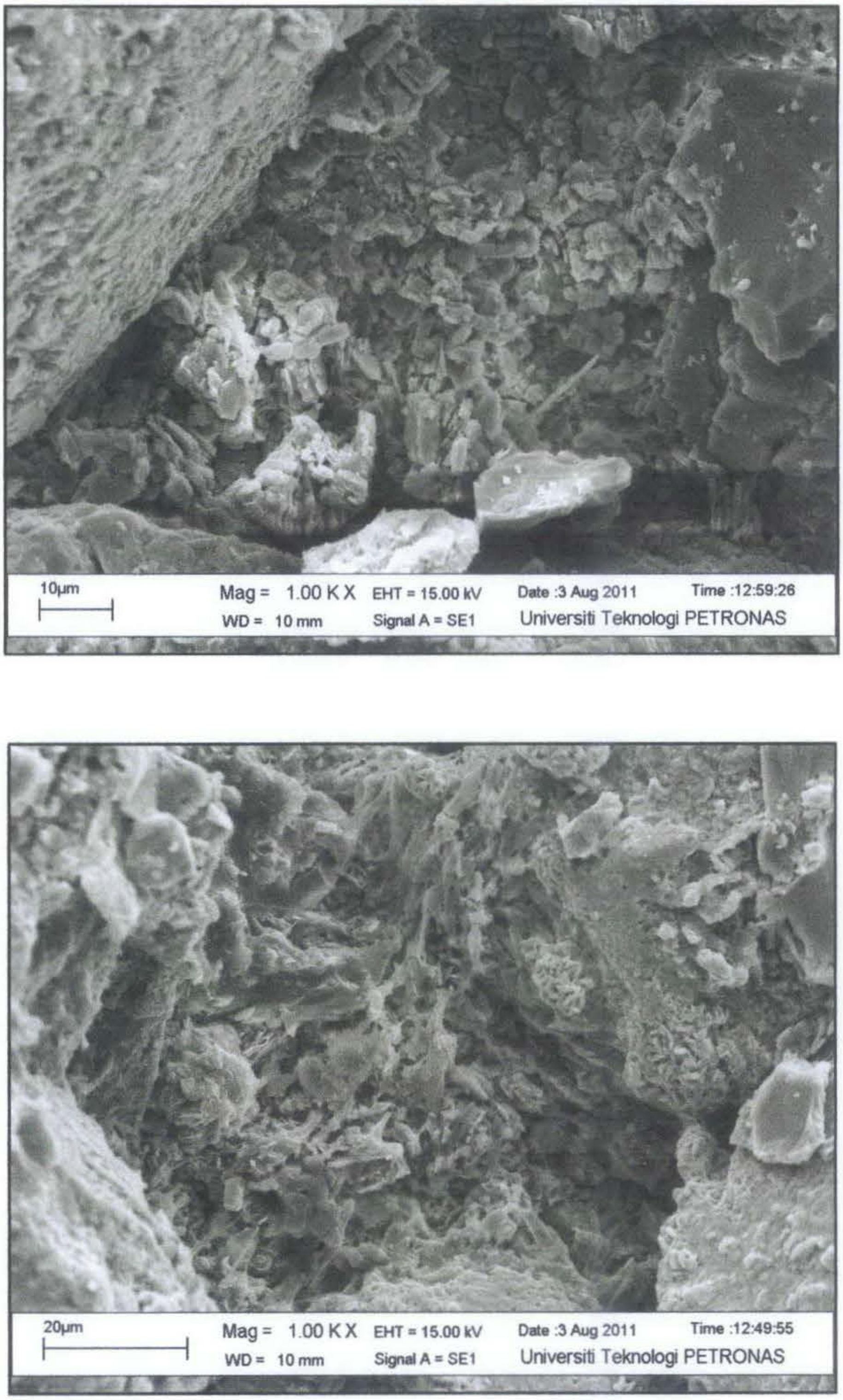


Figure 4.13: Sample 3 precipitates morphology using SEM

Figure 4.13 shows precipitates morphology where from here we can observe that crystals are not clearly seen through the pore throats. However, the precipitation of $BaSO_4$ still can be observed from the SEM images obtained.

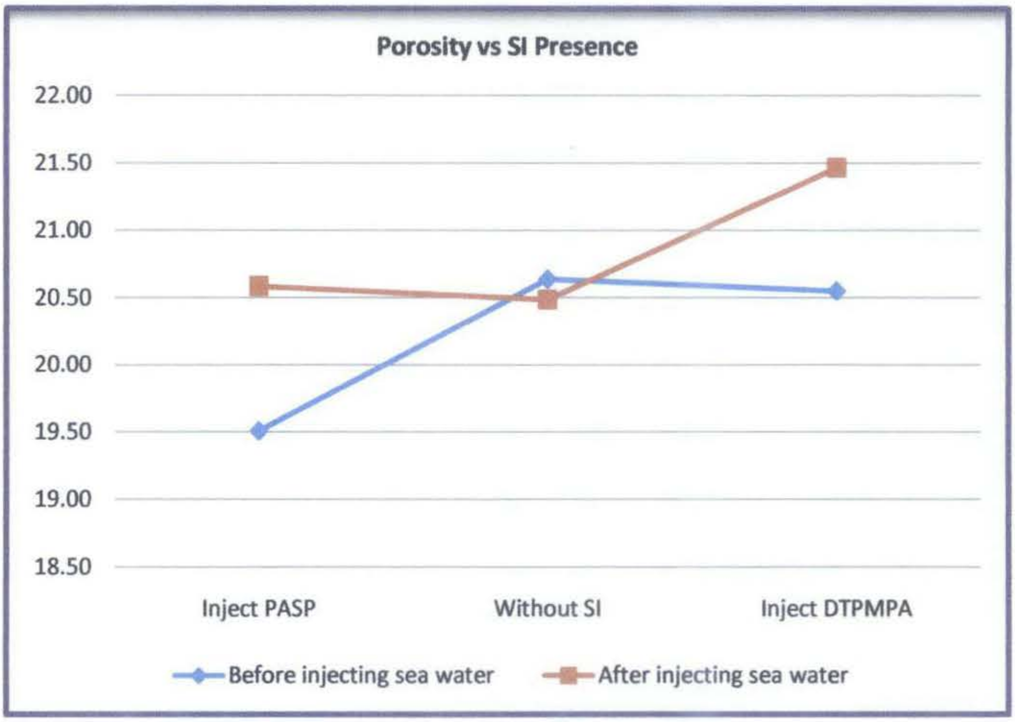


Figure 4.14: Core porosity versus SI presence

Above shows a graph of porosity versus the presence of SI. Based on Figure 4.14, it can be observed that the effective porosity is increased with the presence of the two SIs. These are due to a high SI concentration used (5000ppm) and the SI behavior to remove the existing precipitates. However, PASP SI shows a better performance due to a high effective porosity increment obtained. Effective porosity is reduced without the presence of SI that is caused by $BaSO_4$ scale deposition in porous media.

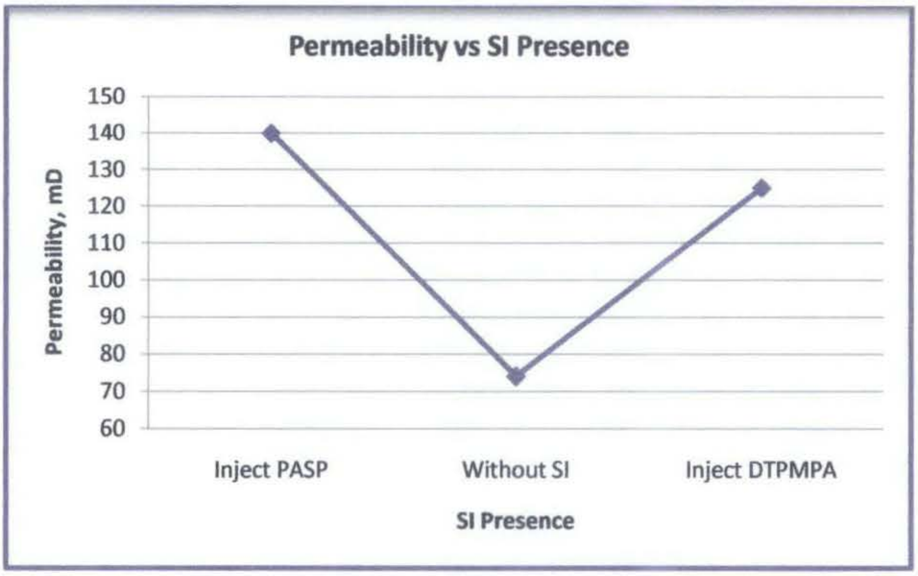


Figure 4.15: Core permeability versus SI presence

Referring to Darcy's Equation (Equation 2.1), permeability (pressure drop is obtained using BPS equipment) can be calculated. Scale precipitation amount varied within this sandstone core whereby more scale near the formation water inlets. Based on Figure 4.15, it is clearly seen that PASP shows a better flow efficiency compared to DTPMPA since it has a higher permeability during Na_2SO_4 injection.

Chapter 5: Conclusion and Recommendation

Conclusion

- Reduced permeability is expected to occur without the presence of scale inhibitor
- Presence of PASP and DTPMPA as a scale inhibitor helps in preventing core plugging
- PASP shows better performance in inhibiting $BaSO_4$ and is competitive enough to replace DTPMPA at this condition (pH=7, Temperature=ambient, SI=5000ppm)
- Higher inhibitor concentration needed to ensure inhibition efficiency

Recommendation

Additional experiments, by varying parameters such as scale inhibitor concentration, pH value and temperature are required before any implementation of this inhibitor in squeeze application. This is to ensure PASP competitiveness as a GSI at different condition.

Chapter 6: References

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