

CHAPTER 1

INTRODUCTION

1.1 Background Study

Oil recovery methods refer to the primary, secondary and tertiary oil recovery. Primary oil recovery is when the reservoir produces naturally without any additional help from fluid or gas injections. Every field will one day loses its natural driving energy to produce oil and gas even though there is still a bountiful amount of it in the reserves to be produced. A matter of fact, some reservoirs are not able to produce naturally in the early stage of its production.

Hence, secondary recovery method is used and it can also be applied simultaneously with primary recovery. Secondary oil recovery method uses conventional methods which are water and immiscible gas injection. Tertiary or enhanced oil recovery uses other methods such as thermal, chemical and miscible injections that can recover oil left in the reservoir after both primary and secondary recovery methods have been exploited. Enhanced oil recovery will help to improve sweep efficiency by decreasing mobility ratio between injected and in-place fluids and eliminate or reduce the capillary and the interfacial forces and therefore improve displacement efficiency (Cârcoană, 1992).

This project will focus on alkaline flooding which is one of the chemical flooding methods. It was recognized early in 1917 by F. Squires that the displacement of oil may be improved by introducing alkali into water (Donaldson, Chilingar et al. 1985). In this method, a high pH chemical is injected whereby if the crude oil contains saponifiable matters, a reaction will occur, forming surfactant in situ. The four mechanism of caustic flooding are emulsification and entrapment, wettability reversal (oil-wet to water wet), wettability reversal (water-wet to oil-wet) and emulsification and entrainment (Johnson Jr., 1976).

In general, formation damage refers to any permeability reduction which will decrease production. According to Doane, Bennion et al. (1999), Formation damage is defined as any type of process which results in reduction of flow capacity of oil, water or gas bearing formation. Alkali such as NaOH, KOH and NaSiO₄ are very effective in mobilizing residual oil in laboratory core floods but it also implies that there will be a high reactivity effects with reservoir rocks which will result in chemical consumption and precipitation of aluminosilicates and thus decreasing permeability (Patino, Civan et al. 2003). Formation damage in alkaline flooding is due to precipitation-dissolution of mineral grains, fines migration, scale formation, pore throat blocking and solid deposition. Hence, this project is intended to investigate if there is any formation damage that will occur due to alkaline flooding.

1.2 Problem Statement

Alkaline flooding has been a preferable method for enhanced oil recovery recently as it is inexpensive and can be readily available. In lab and field applications of alkaline flooding, there have been reports regarding white particles looking like kaolinite which plugged some cores (Hayatdavoudi and Ghalambor, 1996). Besides that, there have been observations in which laboratory studies showed injectivity and permeability damage and scaling as well as plugging problems in the field (Donaldson, Chilingar et al. 1985). Alkali will react with the reservoir rocks and the dissolved materials will eventually plug the pores. Regardless of its ability to mobilize residual oil by emulsification and wettability shift, there is a probability that the fines migration will cause precipitation and thus cause permeability reduction. Besides that, there are also studies on viewing the plugging and precipitation within the pores by visualization but majority of it are inconclusive in terms of how it is interpreted.

At the moment, there are studies regarding formation damage in caustic flooding, however, most focused on a specific mechanism or a certain factor that will cause formation damage. Besides that, studies are done usually in terms of oil recovery and not formation damage. Since there are no specific research done to compare how extent the damage under different conditions, this project will investigate formation damage in alkaline flooding using different alkali in order to get a better understanding of alkaline flooding and optimize its application.

1.3 Objectives and Scope of study

The objectives of this study are as follow:

- To investigate the extent of formation damage in alkaline flooding.
- To visualize the extent of formation damage using Field Emission Scanning Electron Microscope (FESEM)
- To verify formation damage by calculating skin using Hawkins formula

The scope of study involves:

- Determining type of alkaline to be used
- Researching the type of reservoir rock to be tested
- Finding out the factors that will effect formation damage in alkaline flooding
- Studying equations and software for correlation

1.4 Relevancy of the Project

The study will produce experimental results that will indicate the extent formation damage that occur during alkaline flooding. The result will be based on the differential pressure. Hence, a better understanding on alkaline flooding will be achieved for an improved caustic flooding application. Besides that, through the experiment, the skin is calculated to verify formation damage.

1.5 Feasibility of the Project within the Scope and Time frame.

With proper planning and commitment in executing this research, the project should be able to be completed within the 8 months. During FYP 1, research on the theoretical understanding of this project will be done and the design of the experiment, including equipment, chemicals and procedure required will be carefully planned. FYP 2 will focus on execution of the experiments and interpretation of the results obtained. If all materials for the experiments are acquired before FYP 2, it can be conducted earlier during FYP 1. The cost of this project is affordable since the only thing need purchasing would be the chemicals while the equipment is readily available in the university.

CHAPTER 2

LITERATURE REVIEW

In order to have a better understanding of this project, it is important to do some research regarding the mechanism of alkaline flooding, formation damage and its relation.

2.1 Alkaline Flooding and its Mechanism

Alkaline flooding distinguished itself from other techniques as the fundamental basis of the chemicals generated in-situ (Patino, Civan et al., 2003). The alkali injected will react with the organic materials in the reservoir rock itself to produce salt that are surface active. The basic alkaline flooding process starts with a softened water preflush injection followed by the injection of an alkaline solution and by continuous injection of drive water. Polymer slug may be desirable to be injected behind the alkaline solution to control mobility and improve sweep efficiency provided that it is within the economic limits (Cârcoană 1992).

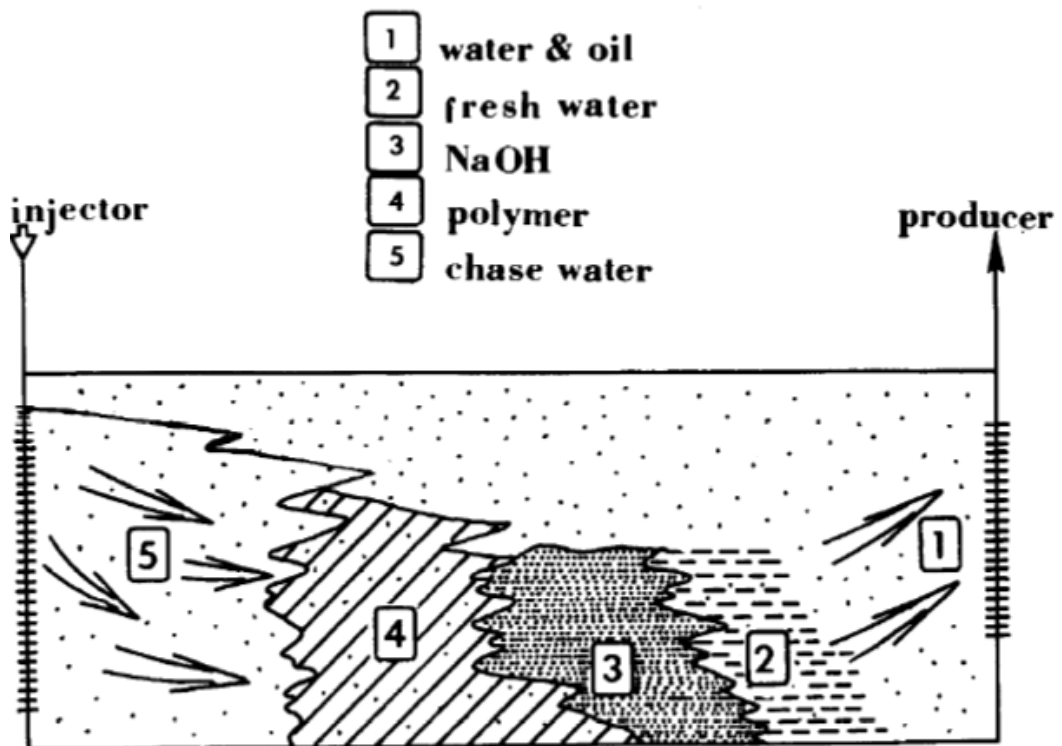


Figure 1: Schematic of Alkaline Flooding Process (Cârcoană 1992).

Based on Johnson Jr. (1976), there are four mechanism of alkaline flooding which are emulsification and entrainment, wettability reversal (oil-wet to water wet), wettability reversal (water-wet to oil-wet) and emulsification and entrapment. Emulsification and entrainment refers to the emulsification of crude oil in-situ and entrained by the flowing aqueous alkali. Wettability reversal for oil-wet to water-wet means oil production increases due to favourable changes in permeability accompanying the change in wettability while water-wet to oil-wet is when low residual oil saturation is attained through low interfacial tension and viscous water-in-oil emulsions working together to produce high viscous capillary number. Emulsification and entrapment which is proposed by Jennings Jr., Johnson Jr. et al. (1974) suggest that if the interfacial tension is low enough, residual oil could be emulsified and move downstream with the flowing caustic and could be entrapped again by pore throats that are too small for the oil emulsion droplets to penetrate.

2.2 Alkali Fluid Interaction with Crude oil and Rock

Alkali flooding method involves a chemical reaction between chemicals of alkali agents and organic (naphthenic) acids in crude oil to produce surfactants (soaps) that can lower the interfacial tension. Alkalis used for caustic flooding are usually sodium hydroxide, sodium carbonate, sodium orthosilicate, sodium tripolyphosphate, sodium metaborate, ammonium hydroxide and ammonium carbonate (Sheng, 2010). When alkali dissociate, it will give a high pH. NaOH dissociates to produce OH^- :



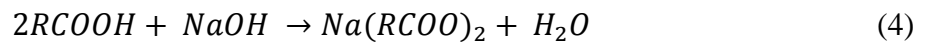
While sodium carbonate will dissociate to produce CO_3^{2-} :



Followed by a hydrolysis reaction:



Naphthenic acid is the name for an unspecific mixture of several cyclopentyl and cyclohexyl carboxylic acids. The naphtha fraction of the crude oil that raffinate is oxidized and produces naphthenic acid. The reaction between the naphthenic acid and alkali (NaOH) is as follow:



Or in general:



This hydrogen bonding interaction between the ionized and neutral acids can lead to the formation of complex chemical called acid soaps which reduce the IFT. Hence, lower IFT will result in easier emulsification.

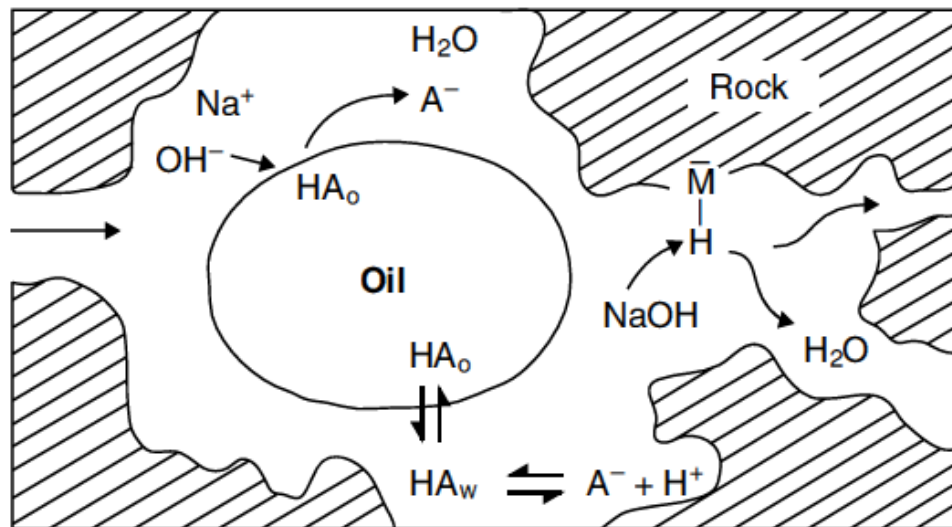
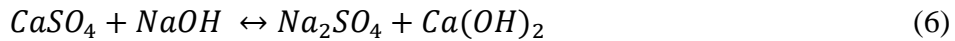


Figure 2: Alkaline recovery process. (deZabala, Vislocky et al., 1982)

Alkali and rock interaction is complicated because of the complex mineralogy in the reservoir. Clay play an important role in alkaline displacement process whereby hydrogen ions in clay will react with hydroxide ions in the flood which consume the alkaline solution as it moves through the reservoir. This goes to

calcium ions as well as it will exchange for sodium ions in the alkaline solution (Sheng, 2010). Example of alkali reaction with rock is:



According to Bagci, Kok et al. 2001, in the alkali flood experiments, reduction in permeability value and plugged pores is observed at the interface of high pH solutions with formation. Formation damage is a general terminology referring to the impairment of the permeability of petroleum bearing formations by various adverse processes (Moghadasi, Jamialahmadi et al., 2004). The two main mechanism of formation damage in alkaline flooding is the effect of fines migration and scale precipitation (Patino, Civan et al., 2003). Strong alkali are very effective for mobilizing residual oil in lab core floods but implies also higher reactivity effects with reservoir rocks (clays, basically kaolinite) leading to chemical consumption and precipitation of aluminosilicates and thus leading to a decrease in the value of permeability.

2.3 Factors That Affect Alkaline Flooding

There are several factors that affect alkaline flooding. These factors are the oil acid number, alkaline concentration and type, brine salinity and temperature. Based on Ge, Feng et al. (2012) paper, it can be concluded that heavy oil demonstrate incremental oil recovery increases with the oil acid number, which is a prominent factor for alkaline flooding. A poor sweep efficiency is shown when the alkaline concentration is low. The brine salinity can change the created emulsion type and influence its properties, which is related to the displacement efficiency.

The relatively low temperature is actually beneficial to alkaline flooding. Therefore, it can be said that when the temperature increases, the displacement efficiency declines intensively. Consumption of the alkali increases with increasing temperature and decreasing flood rate (Mehdizadeh and Handy, 1989). Therefore, higher temperature may formation damage in alkaline

flooding as most reactivity of alkaline and rocks in formation are accelerated in higher temperature.

2.4 Effects of Using Different Alkaline

Studies showed that by using different alkaline will give different results as different alkaline components used will behave differently in the experiment. Campbell and Krumrine (1979) used sodium orthosilicate and sodium hydroxide to test for its efficiency in alkaline flooding and showed that sodium orthosilicate gives higher oil recovery than sodium hydroxide under the same conditions.

In Ge, Feng et al. (2012), two different alkaline were used to investigate the incremental oil recovery as a function of alkaline type. It is stated that the incremental oil recovery of sodium hydroxide for Zhuangxi heavy oil is always higher than sodium carbonate under the same conditions. Hence, the alkaline type is one of the important factors that should be considered in alkaline flooding.

2.5 Formation Damage Determination

Formation damage is normally determined in terms of skin. Skin is a dimensionless factor which explains a zone has enhanced or reduced permeability. A positive skin would indicate that there is permeability impairment while negative skin indicates enhanced permeability, usually due to stimulation. Skin factor, s , can be calculated using Hawkins formula (Civan, 2007).

$$s = \left(\frac{k}{k_d} - 1 \right) \ln \left(\frac{r_d}{r_w} \right) \quad (7)$$

One of the methods in determining the formation damage is by conducting a core flooding experiment. From the laboratory test, the permeability reduction

can be determined and a plot of the damage ratio with time can be plotted (Bin Merdhah A.B., Mohd Yassin A.A., 2007).

Damage Ratio (DR) is given by:

$$DR = \frac{k_d}{k_i} = \frac{\text{rock permeability after damage}}{\text{Original rock permeability}} \quad (8)$$

Where the permeability can be determined by Darcy's Law:

$$k = \frac{Q\mu L}{\Delta P A} \quad (9)$$

Where,

k = permeability (Darcy)

μ = liquid viscosity (cp)

Q = flow rate (cc/sec)

L = length of core (cm)

ΔP = differential pressure across core holder (atm)

A = cross-sectional area of core (cm²)

Based on Patino, Civan et al. (2003), diagnostic equations developed by Wojtanowicz et. al. and Civan provide a practical and rapid means of determination of the governing formation damage mechanisms. The Wojtanowicz et. al. model identified the formation damage mechanisms as pore surface deposition and sweeping while Civan's model is better suited for permeability variation due to scale dissolution and precipitation. Civan derived a new model for variation permeability reduction index with time:

$$\frac{K}{K_0} = (1 + \lambda t)^{-\delta} \quad (10)$$

Where,

K = absolute permeability, md

K₀ = initial absolute permeability, md

λ = lumped parameter

t = time, min

δ = lumped parameter

2.6 Visualization Methods

Some visualization methods have also been suggested by van der Zwaag, Stallmach et al. (1997) to investigate formation damage. The methods are by using Scanning Electron Microscopy, Computed Tomography (CT) Scanning and Nuclear Magnetic Resonance (NMR) Imaging. From these visualizations methods, the pre-flooded pores of the core can be compared with the post-flooded core in order to see if there is any graphic indication of any sort of precipitation in the core.

CT Scanning is a non-destructive imaging technique that uses X-ray technology and mathematical reconstruction algorithms to view cross sectional slices of an object (Siddiqui, S. & Khamees, A. A., 2005). It has been used in the industry for quite some time in order to obtain images on core sample. The CT scan is used for two main reasons which are for core description and fluid flow characterization (Bataweel, Nasr-El-Din et al. 2011). Most CT scanners can handle large samples however they have limited resolution for measuring density and porosity.

NMR imaging measurements are based on the surface and bulk interactions of the rock with any hydrogen containing fluid. These measured values are classified by the time-independent relaxation behaviour of the NMR signal (Funk, J. et al., 2000). It can be used for analysing whole cores. Nevertheless, smaller pore sizes may be undetected with NMR and more tests are required to optimize the parameters to identify the smaller pore sizes.

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface

topography, composition and other properties such as electrical conductivity (K.R., 2008). Besides that, Bonnie and Fens (1992), stated that the images of the rock sample can develop a procedure in order to derive porosity and permeability data. The flexible and non-destructive structure of this tool makes it suitable for analysing cuttings, however, can handle small sample in which the core must be cut to a smaller pieces.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

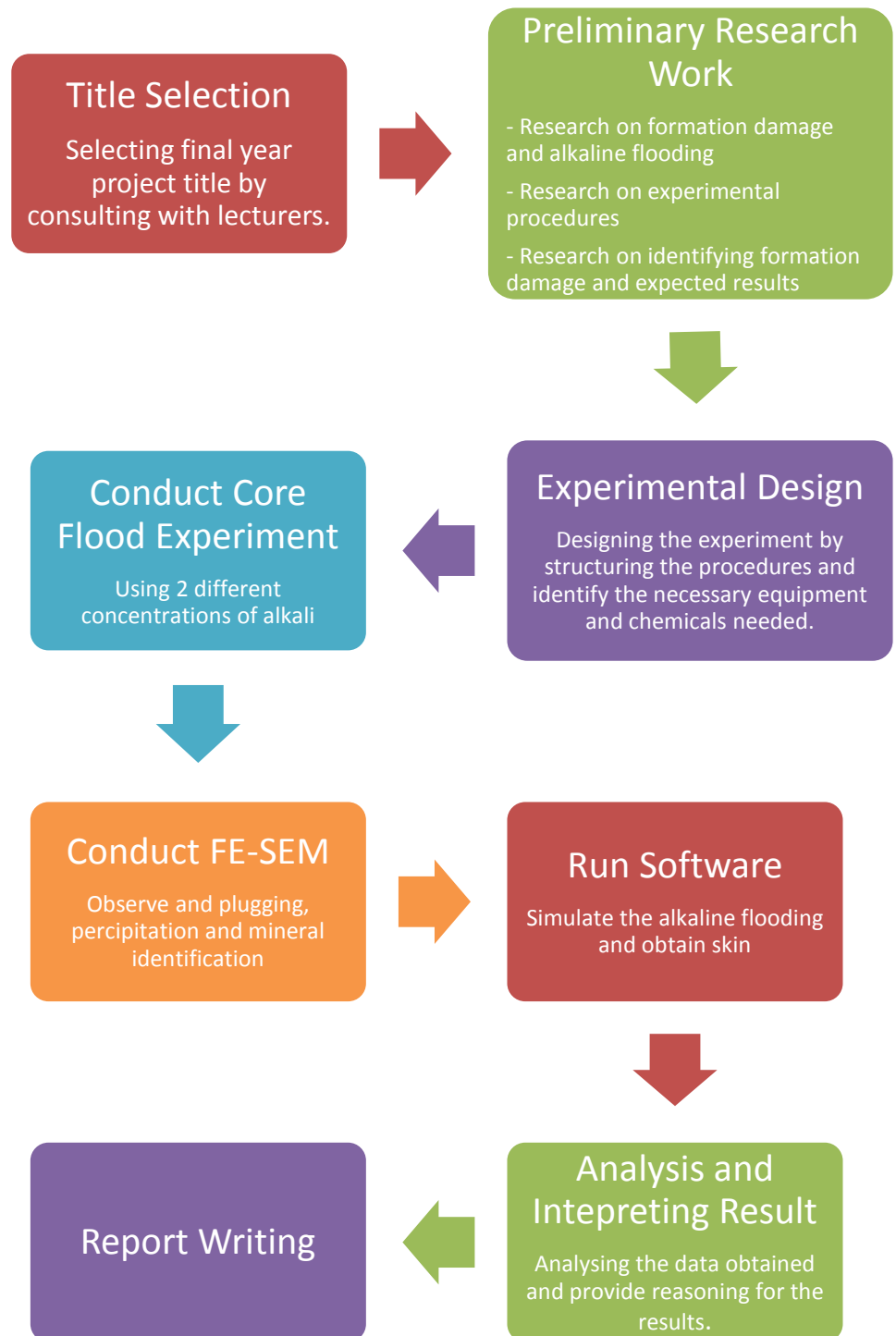


Figure 3: Research Methodology Flow Chart

| ACTIVITIES | WEEK | | | | | | | | | | | | | |
|---|------|---|---|---|---|---|-----------|---|---|----|----|----|----|----|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| Project Work Continues | █ | █ | █ | █ | █ | █ | SEM BREAK | | | | | | | |
| - Conduct experiment | █ | █ | █ | █ | █ | | | | | | | | | |
| - Result discussion and interpretation | | | | | █ | █ | | | | | | | | |
| Submission of Progress Report | | | | | | █ | | | | | | | | |
| Project Work Continues | | | | | | | | █ | █ | █ | █ | █ | | |
| - Result discussion and interpretation | | | | | | | | █ | █ | █ | █ | █ | | |
| Pre-SEDEX | | | | | | | | | | █ | | | | |
| Submission of Draft Report | | | | | | | | | | | █ | | | |
| Submission of Dissertation (soft bound) | | | | | | | | | | | | █ | | |
| Submission of Technical Paper | | | | | | | | | | | | █ | | |
| Oral Presentation | | | | | | | | | | | | | █ | |
| Submission of Project Dissertation (hard bound) | | | | | | | | | | | | | | █ |

Figure 5: Gantt chart for FYP 2

3.3 Experimental Methodology

3.3.1 Compatibility Test

Materials: Sodium hydroxide, sodium chloride, distilled water, crude oil

Apparatus: Test tube, measuring, cylinder, electronic balance, magnetic stirrer

Procedure:

1. The mass of sodium hydroxide is calculated by using the formula below
Weight percentage (w/v) = [Mass of solute (g) / Volume of solution (ml)] x 100
Example: 0.5% = [0.025g / 10 ml] x 100
2. The mass of sodium hydroxide is weighed on the electronic balance.
3. 5ml of distilled water is poured into the test tube and followed by adding the sodium hydroxide.
4. Stir the mixture using a magnetic stirrer until it dissolves completely.
5. Step 1 till 4 is done for preparing 5ml of 1% wt sodium chloride as well.
6. The sodium chloride is added into the test tube with sodium hydroxide
7. About 5ml of crude oil is measured and added into the test tube and shake for several minutes
8. The test tube is then kept in the oven.
9. Observe if there is any precipitation occurred.

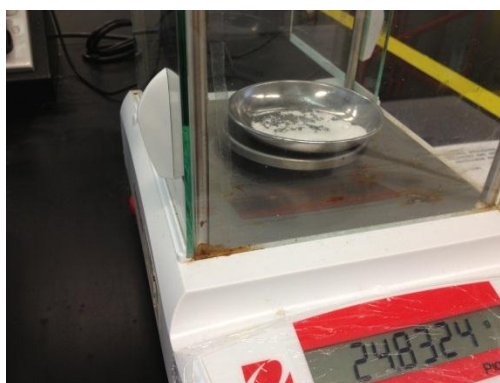


Figure 6: Measuring sodium chloride using electronic balance

3.3.2 Core Cleaning

Materials: Core, Toluene, Carbon Dioxide

Machine: Carbon Dioxide Core Cleaner

Procedure:

1. Switch on fume hood
2. Place the switch to manual position
3. Unscrew the cap of carbon dioxide cleaner and remove the core plug basket from the cell
4. Load sample to be cleaned into the basket and place the basket in the cell
5. Screw the cap on the cell until the threads of the cap bottom out
6. Place switch to automatic mode for long (12 hours)
7. Turn the cooling water on and check the drain water flowing out
8. Press start button to begin the process

After 12 hours, the samples are loaded out and dried in the oven.



Figure 7: Carbon Dioxide Core Cleaner

3.3.3 Porosity and Permeability Determination

Materials: Core

Machine: Measuring caliper, electronic balance, PoroPerm

Procedure:

1. Measure the diameter and length of the core using a measuring caliper and the weight using an electronic balance
2. Input the measurements in the measure tab
3. Load core into the cell
4. Turn on the pressure valve and ensure the confining pressure is about 400psi
5. Press the start button

The porosity and permeability of the cores are measured before and after the core flooding experiment.

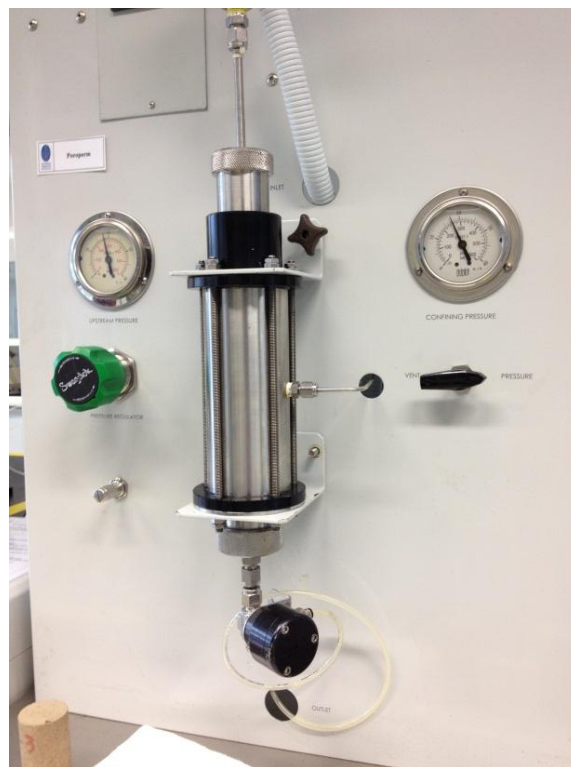


Figure 8: PoroPerm Machine

3.3.4 Core Saturation

Materials: Core, Brine

Apparatus: Beaker

Machine: Vacuum pump

Procedure:

1. Put cores into the glass vacuum pump filled with distilled water
2. Close the glass ballast ensuring that the inlet valve is connected to the pump

3. Turn the switch on
4. Let it run for 6 hours
5. After 6 hours, close pump.
6. Remove cores and put it into a beaker filled with 1% NaCl

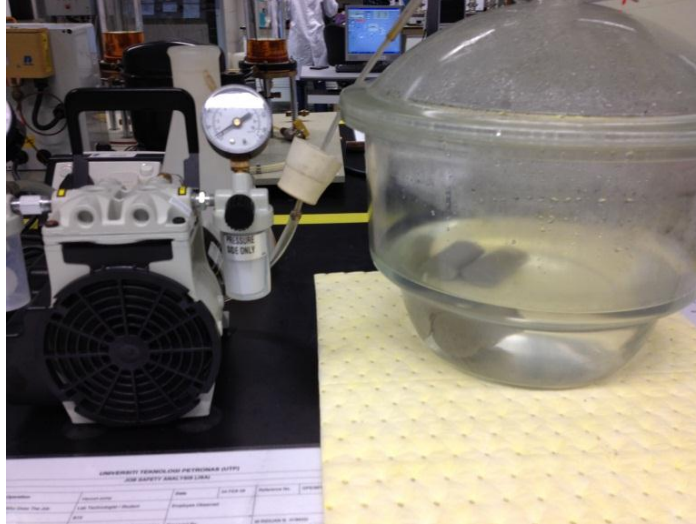


Figure 9: Vacuum pump

3.3.5 Core Flooding

Materials: Brine, Dulang Crude Oil, 0.5% NaOH, 1.5% NaOH

Apparatus: Graduated cylinder

Machine: Relative Permeability System

Procedure:

The core flood will start with pre-flush brine in which the core permeability to brine is obtained with a constant pump rate. It will then be flooded with crude oil until it reaches the irreducible water saturation. Brine is the flooded again and displaced the oil. This process will stop once there is no oil observed in the effluent. This is then followed by a continuous flooding of alkaline in order to remove the residual oil. The alkaline flooding run is conducted using the same oil with two different alkaline concentrations.

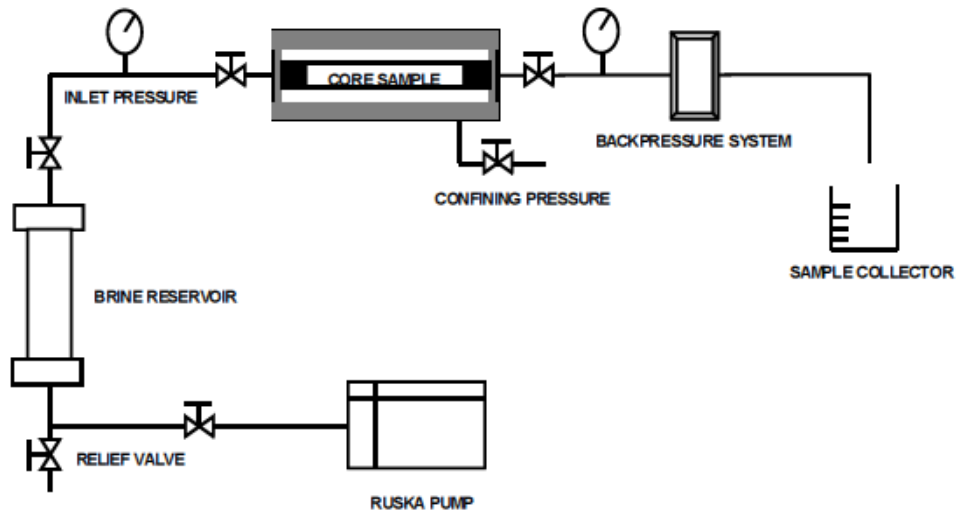


Figure 10: Schematic for core flood experiment.

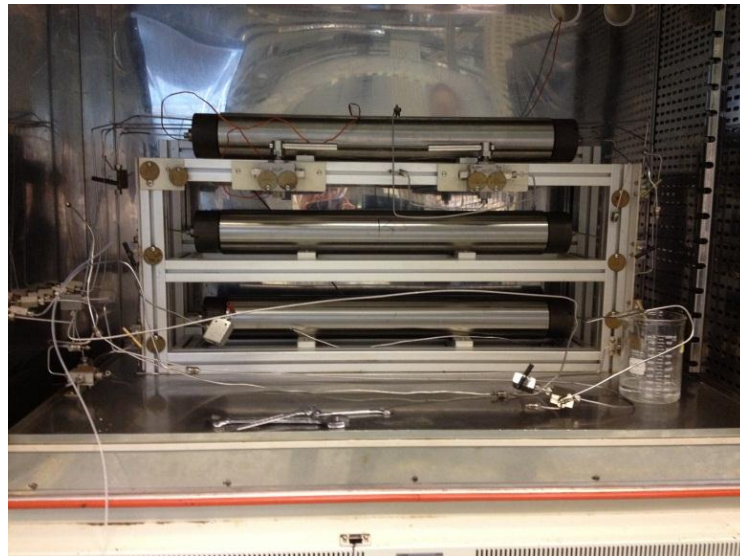


Figure 11: Core Flooding Relative Permeability System

3.3.6 Field Emission Scanning Electron Microscope

Materials: Core

Machine: FE-SEM

Procedure:

1. Sample preparation

Thin slice of core samples are obtained before and after the alkaline flooding experiment

2. Mineral Identification

The SEM consists of energy dispersive x-ray (EDX) which has the capability in identifying the minerals in the sample.

3. Results Comparison

The different in mineral composition in the samples are compared. This will allow us to see if there are any precipitations formed due to alkaline reactivity with the rocks. Besides that, the location of the new minerals will be identified to see if it causes plugging in the rocks.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Compatibility Test

The result for this experiment is presented in Figure 12 and 13 whereby the alkaline is of different concentrations. It is also noted that the Dulang crude oil used has an acid number of 0.93 mg KOH/g oil. From the result, it is observed that all 4 test tubes have no precipitation. According to Elraies and Tan (2010), if there is white precipitation observed after mixing the alkaline, brine and crude oil, the alkaline concentration is not compatible. Therefore, since no cloudy or white precipitation seen in any of the test tubes, all 4 alkaline concentrations are compatible with 1% wt of sodium chloride and crude oil. Besides that, it would suggest that during core flooding, there should be a minimal fluid-fluid interaction that would have caused formation damage in the core.

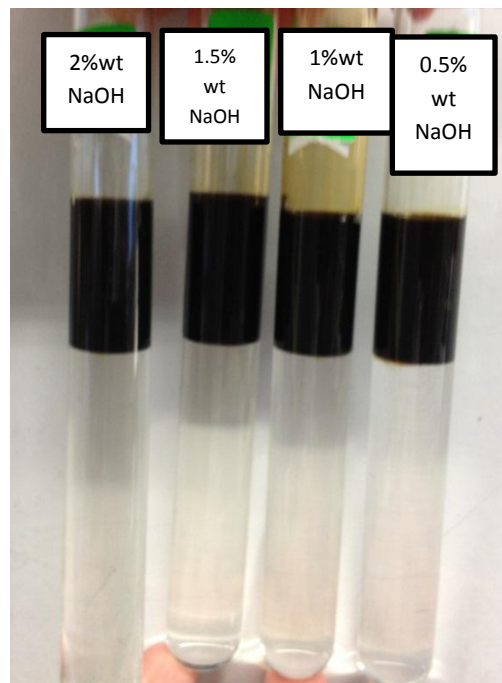


Figure 12: At the beginning of compatibility test

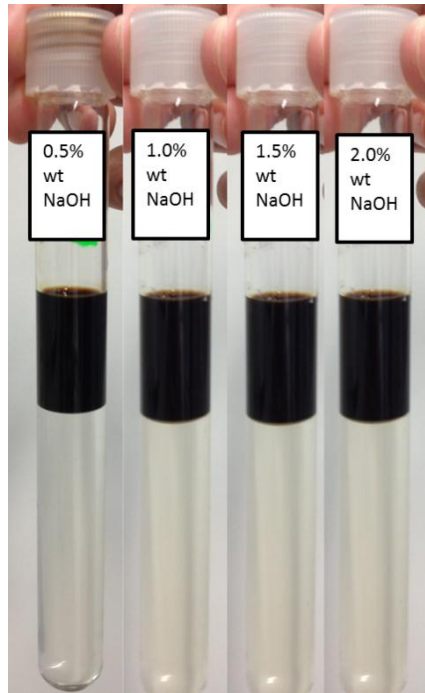


Figure 13: Compatibility test after 7 days.

4.2 Core Flooding Experiment

Based on the compatibility test, since all four concentrations seems to be compatible, two different concentration of sodium hydroxide is used to investigate the formation damage in alkaline flooding.

From this experiment the initial and end permeability of the rocks were obtained using poroperm machine. The results of the permeability are shown in table 1. The differences between the two permeability shows whether there are any increase or reduction in the rock permeability. For core 1, we can see that the decrease in permeability is immense with 62.28% and for core 2 with 37.62%. This suggests that reaction between the alkaline with rock, brine and crude oil has definitely occurred and causes precipitation within the rock.

When comparing the permeability reduction, 0.5% NaOH causes more damage which suggests that reaction has occur within the core which reduces the interconnected pores within the rock. Using 0.5% NaOH would give more reaction with the crude oil

and the rock itself to form a precipitate such as Na_2SO_4 and $Ca(OH)_2$ as stated mentioned in the literature review due to the reaction of alkali with clay in the rocks. This new minerals form will plug the pores that are interconnected causing the permeability to greatly decrease.

| Sample | 0.5% NaOH Core 1 | 1.5% NaOH Core 2 |
|---------------------------|---------------------|---------------------|
| Initial permeability (md) | 52.38 | 33.90 |
| end permeability (md) | 19.76 | 21.14 |
| Permeability Reduction | 62.28% | 37.62% |

Table 1: Permeability Reduction

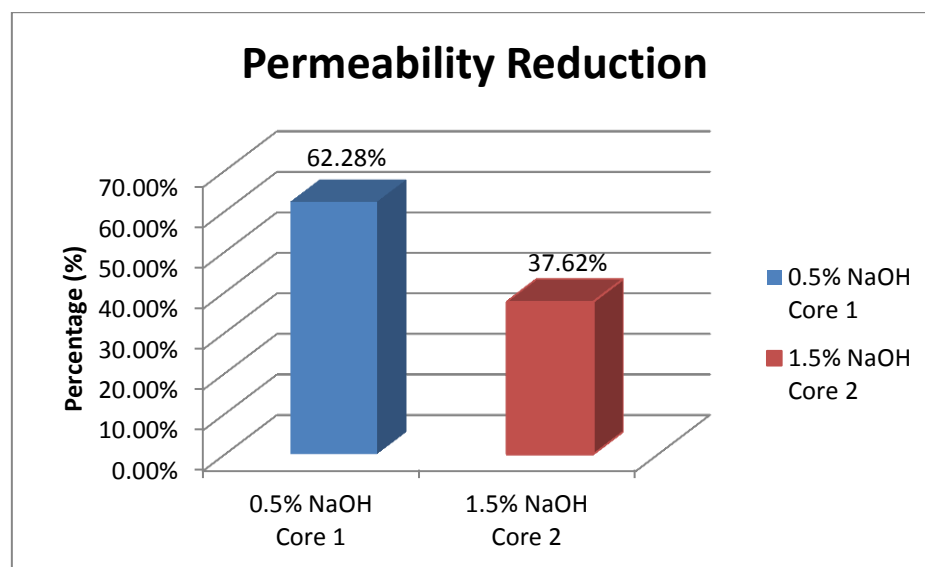


Figure 14: Graph of Permeability Reduction Percentage

From the differential distribution of the core flooding experiment in figure 15 and 16, we can see a significant increase in delta pressure which indicates that precipitation had occurred during the alkaline flooding. For core 1, during core flooding, there is a steady increase in pressure difference from min 28 till min 69 which ranges from 40 to 51 psig. This is quite high as the previous water flood pressure differential ranges from 10 to 30 psig. The increase in the delta pressure is due to increase in precipitation occurring in the rock itself hence damaging the core by reducing its

permeability. After min 69, there was a short circuit that occurred in the lab causing no electricity to run the machine for about 20 minutes. After starting up again the machine, it can be seen that differential pressure drops since no fluid was injected and starts building up again after a while. The next significant pressure drop was due to a blackout which had affected the whole campus. The machine was only able to run almost 2 hours after the incident. However, we could still see the increase in differential pressure towards the end of the run.

There were no disturbances during the second run hence we can see the pressure differential distribution for core 2 quite clearly (figure 16). The increase in pressure difference can be seen from min 68 to min 156 and ranges from 52 to 69 psig. This indicates that precipitation also occurred during the core flooding.

Table 2 and 3 shows the oil recovery factor during water flooding and alkaline flooding. Comparing the incremental recovery of both runs, core 1 gives 27.34% increase in recovery while core 2 with a 27.78% increase. Hence, core 2 which uses 1.5% NaOH can be said to give a slightly higher recovery than core 2 which uses 0.5% NaOH. The reason why core 1 produces a lower incremental recovery percentage regardless of a higher initial permeability is supported by the higher permeability reduction.

During water flooding, the brine injected into the core would also have cause damage within the core. It is important to note that chemical consumption also occur during alkaline flooding in which the alkali dissolve rock minerals such as aluminium, silica and oxygen (aluminosilicates). The minerals formed are also referred to as scales which are not desirable in any producing wells. The dissolution reaction between alkali and rock is the reason why a reasonable increase in recovery during the tertiary recovery is observed. However, the rate of precipitation may be greater than the rate of dissolution resulting in a decrease in permeability.

Figure 17 to 19 shows the effluent obtained for run 1 of 0.5% NaOH while Figure 20 to 22 shows the effluent for run 2 of 1.5% NaOH. It is from the effluent that we can get the volume of oil displaced during the water and alkaline flooding.

| CORE 1 (0.5% NaOH) | |
|---|--------|
| Original Oil In Place (ml) | 12.80 |
| Volume Displaced oil after water flooding (ml) | 6.20 |
| Volume Displaced oil after alkaline flooding (ml) | 3.50 |
| Recovery factor for water flooding (%) | 48.44% |
| Recovery factor for alkaline flooding (%) | 75.78% |
| Incremental recovery (%) | 27.34% |

Table 2: Volume of oil and water displaced and recovery factor for Core 1

| CORE 2 (1.5% NaOH) | |
|---|--------|
| Original Oil In Place (ml) | 9.00 |
| Volume Displaced oil after water flooding (ml) | 6.00 |
| Volume Displaced oil after alkaline flooding (ml) | 2.50 |
| Recovery factor for water flooding (%) | 66.67% |
| Recovery factor for alkaline flooding (%) | 94.44% |
| Incremental recovery (%) | 27.78% |

Table 3: Volume of oil and water displaced and recovery factor for Core 2

Delta Pressure Vs Time

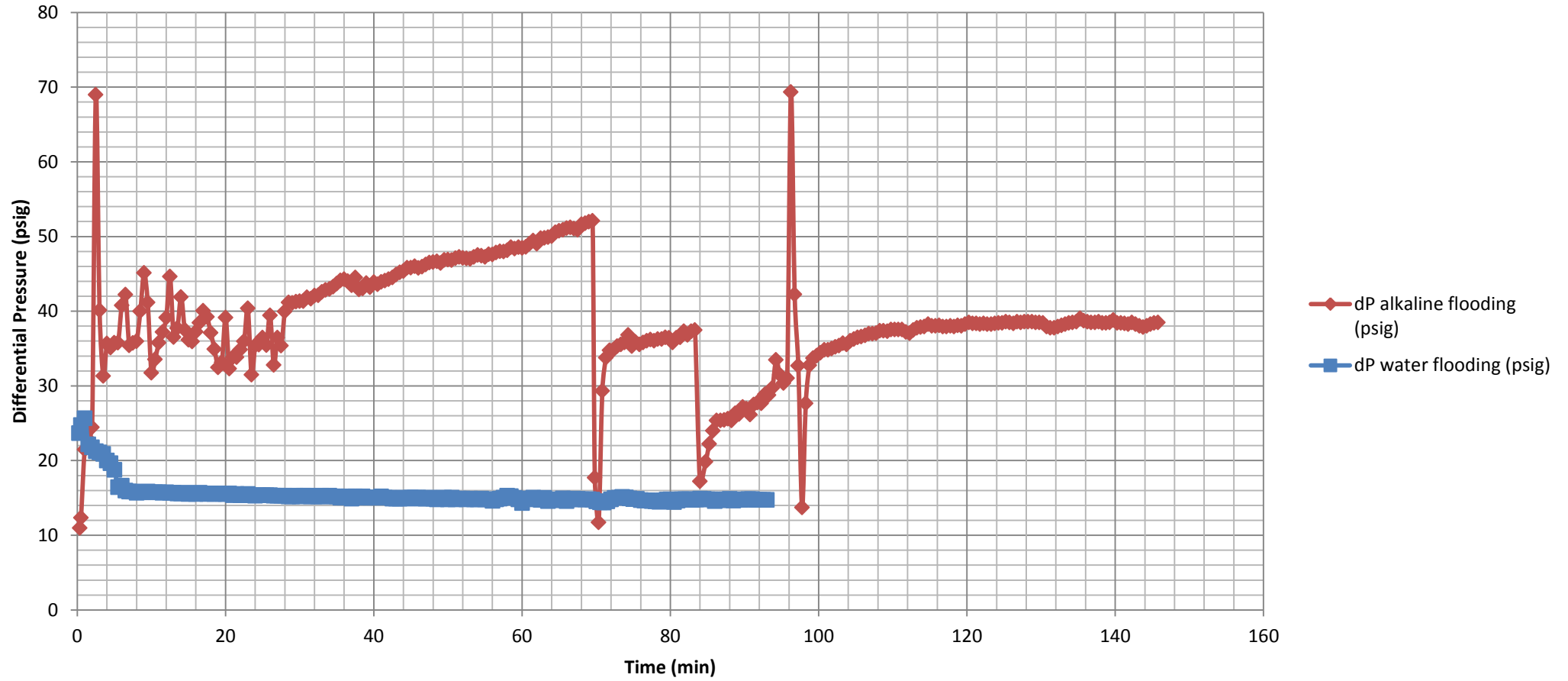


Figure 15: Pressure Differential Vs time for Core 1

Delta Pressure Vs Time

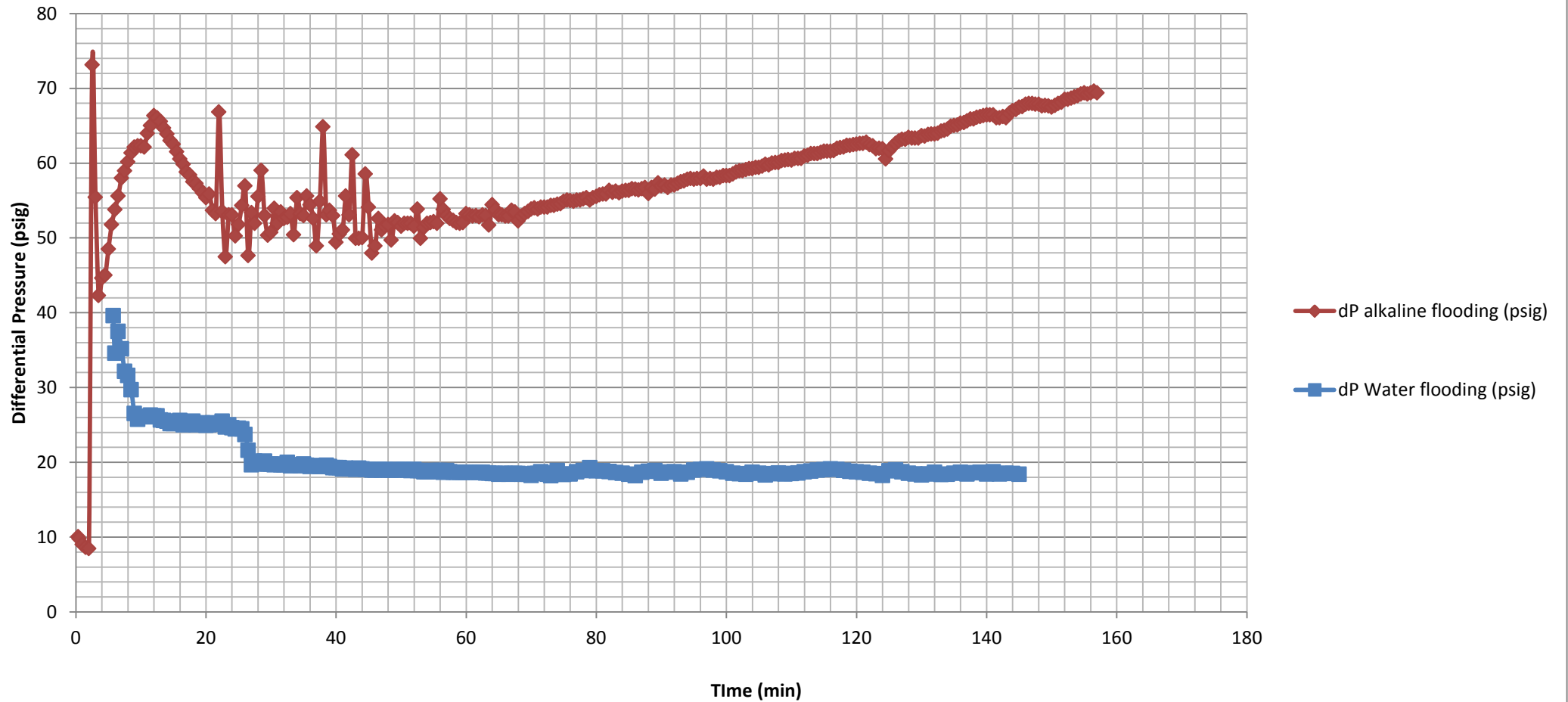


Figure 16: Differential Pressure Vs Time for core 2



Figure 17: Effluent from oil saturate (0.5% NaOH)

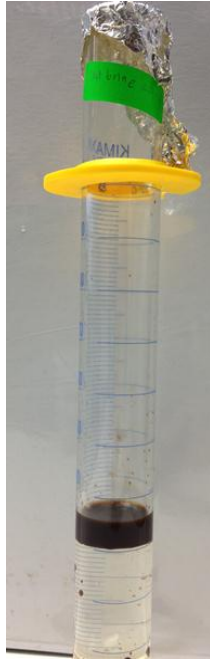


Figure 18: Effluent from water flooding (0.5% NaOH)



Figure 19: Effluent from alkaline flooding (0.5% NaOH)

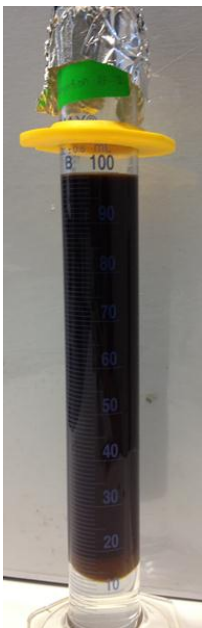


Figure 20: Effluent from oil saturate (1.5% NaOH)



Figure 21: Effluent from water flooding (1.5% NaOH)

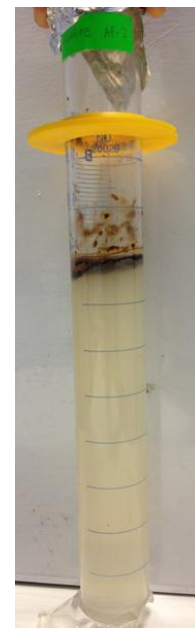


Figure 22: Effluent from alkaline flooding (1.5% NaOH)

4.3 Field Emission Scanning Electron Microscopy (FESEM)

The Berea sandstone core sample was analysed using FESEM in order to understand the grain and pore structure. The SEM image is able to provide a three dimensional view of the core sample at a microscopic level. Based on Power's scale, the roundness of the grains for the core sample as shown in Figure 23 and 24 are ranged from subrounded to subangular.

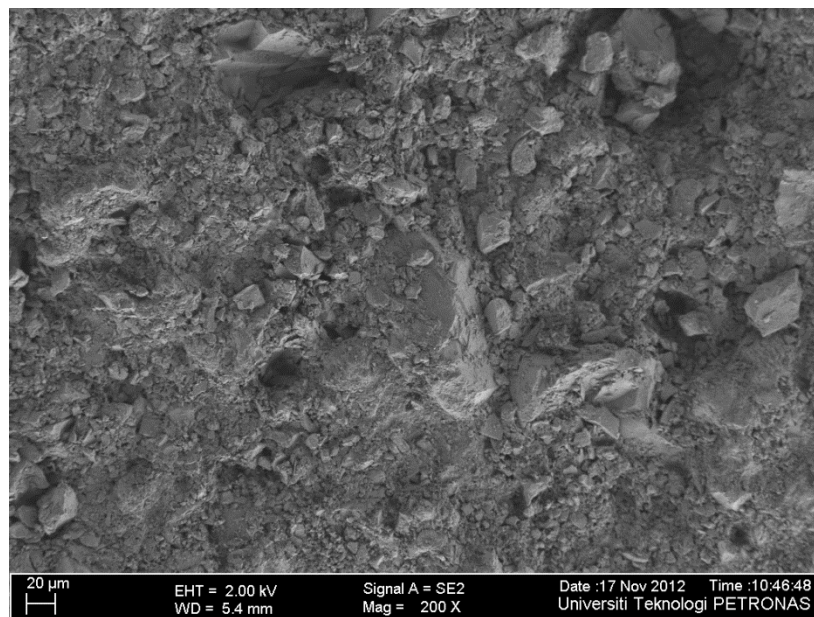


Figure 23: SEM image of clean core sample at 200x magnification

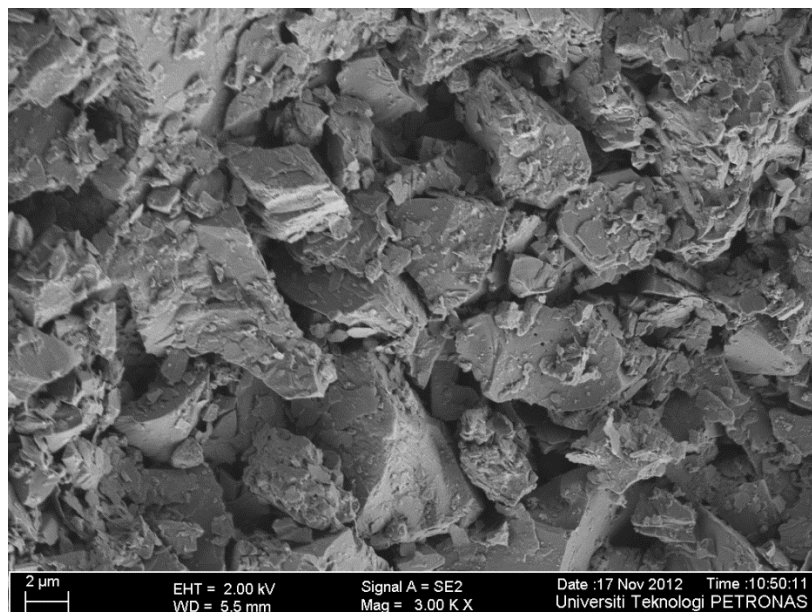


Figure 24: SEM image of clean core sample at 3000x magnification

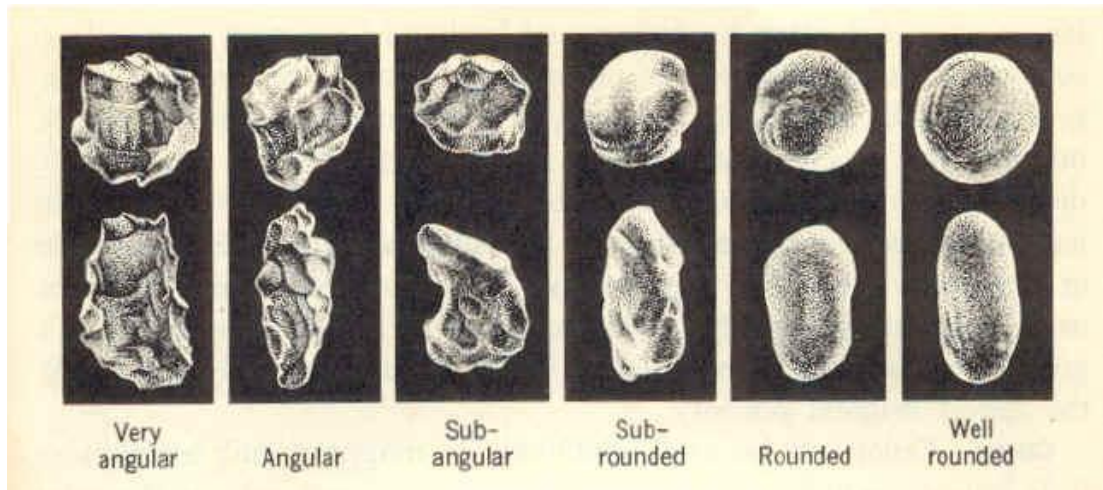


Figure 25: Power's scale of roundness chart (Bergslien 2012)

It is also noted that the porous area of the sample can clearly be seen especially with 3000x magnification as seen in Figure 24. The pores' diameters are generally of 2 μm . According to the EDX analysis of the core sample, the sample has a high content of quartz which is normal in sandstone and similar to the many other researches done on Berea sandstone such as Carr & Paschke (1998) and Mohan, K. Krishna. et. al. (1993).

The SEM images of core 1 and core 2 after the core flooding experiment is shown in Figure 26 and Figure 27. Both show that there are not many porous spaces as what we can see in the clean core sample (Figure 24). Since the sample was taken only from the inlet and only a few images was obtained from SEM, it is hard to exactly confirm if there is any sort of structural damage such as build up precipitation on the core.

However, the most significant difference that can be seen is a sugary reflection substance that seems to coat certain areas of the rock. The coating could be fine crystallization of iron oxides or aluminium oxides. Scales that are seen in the figures could be due to brine or alkaline or both. Regardless, from the figures, it seems to cover the pores of the core. Although with only certain areas of images covered by SEM, it is not entirely confirmed whether it is pore surface deposition or it is causing partial dissolution in the cores. This layer of mineral suggests that it could be the reason why there are permeability impairments on both cores.

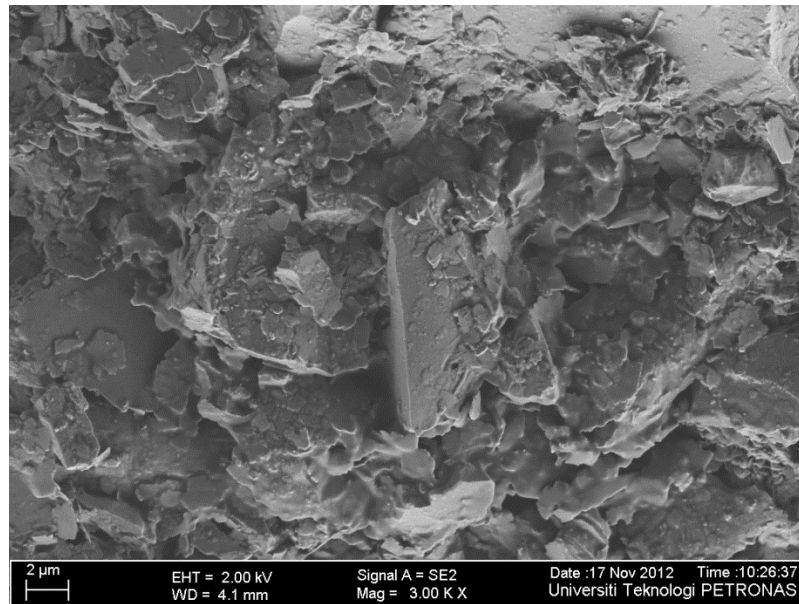


Figure 26: SEM image of core 1 after core flood.

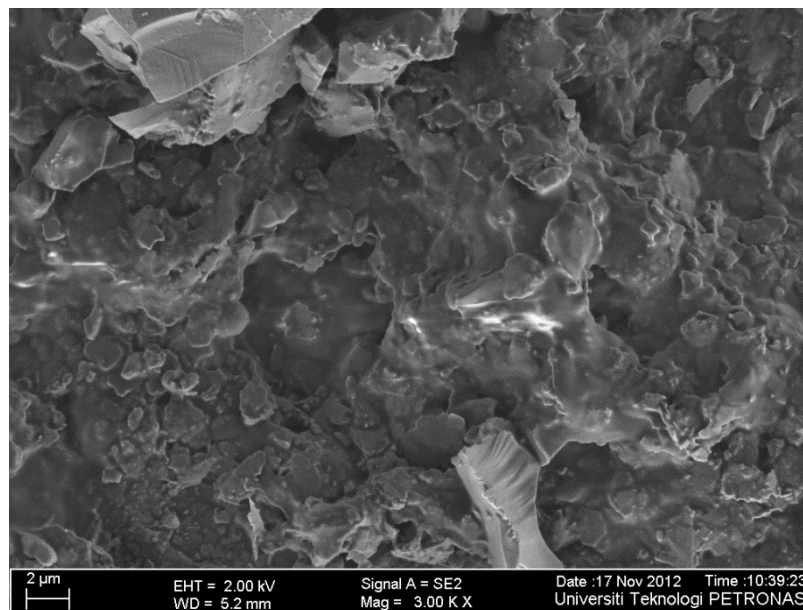


Figure 27: SEM image of core 2 after core flood.

4.4 Skin Calculation

By using Hawkins's formula in equation (7), the skin can be calculated. In the field, a well test or a transient test is done to estimate the extent of damage near the wellbore. For this case, the wellbore radius is assumed to be 0.328 ft while the radius of the damage zone is 3.328 ft. The skin obtained is shown in table 4. The positive skin confirms that there is damage and this indicates that there is a flow rate restriction from the reservoir into the wellbore. In order to improve the flow rate, the damage has to be removed or reduced by increasing the permeability through acidizing or fracturing.

| Sample | Skin |
|--------|------|
| Core 1 | 3.83 |
| Core 2 | 1.4 |

Table 4: Skin factor for core 1 and core 2

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Based on the experiments performed, the compatibility test shows that the alkaline, brine and crude oil used are compatible with each other hence there should be minimal fluid-fluid reaction during the core flooding experiment. Alkaline flooding using 0.5% wt NaOH gives a higher permeability reduction with 62.28% while 1.5% wt NaOH causes permeability to be reduced by 37.62%. A significant reduction in permeability shows that there has definitely been damage in the core. Besides that, the increase in differential pressure indicates that precipitation had happened during the alkaline flooding. Both concentrations of NaOH gives a reasonable incremental of oil recovery which suggests that surfactant is produced and the process of mineral dissolution has also occurred.

SEM images suggest that the formation damage experienced by the core is due to surface deposition which blocks the pore. This is due to the fluid/rock reaction that occurs during alkaline flooding. There has also seems to be fewer distinct images of porous spaces in the cores. The skin calculated gives a positive value which confirms formation damage. 0.5% wt NaOH gives a higher skin value which is justifiable with the higher permeability reduction.

As a conclusion, the objectives of this project which is study the extent of formation damage by analysing the permeability reduction, visualizing the damage using FESEM and determining the skin have been achieved. Therefore, the project is successfully completed.

5.2 Recommendation

There are several improvements that can be made to this project. While conducting this project there has been several issues encountered such as limiting the number of run to two per students when using the core flood machine. Therefore, only one different factor was able to be tested which is using different concentration of alkali. Therefore, in the future, other factors could be tested as well using different alkali, temperatures, pressures, pH and flow rate.

In the future, it is also best to conduct an alkali consumption or absorption rate test. This will help to identify the capability of the core sample itself to consume alkali as it plays a huge role in the alkaline flooding mechanism and may help justify the presence of formation damage or even the efficiency of alkaline flooding.

Besides that, it is also recommended that for the compatibility test to be done for different brine of different salinity as well. Since in different reservoir, brine gives different composition, it would be best to try different brines in order to get a better result and comparison.

One of the challenges encountered was when using the FESEM where only two samples were allowed. Hence, only a few images were able to be obtained and this results in variety of interpretations when viewing the cores. Besides that, the minerals identification should also be done for each image taken by SEM.

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APPENDICES

1. Properties of core

| Description | Core 1 | Core 2 |
|---------------------------------|---------|---------|
| Specimen Length, L (mm) | 75.580 | 76.310 |
| Specimen Diameter, Do (mm) | 38.220 | 38.180 |
| Specimen weight (g) | 187.100 | 190.660 |
| Pressure confining (psig) | 400.000 | 400.000 |
| Sample porosity \emptyset (%) | 16.595 | 17.361 |
| Pore Volume Vp (cc) | 14.368 | 15.145 |
| Sample bulk volume Vb (cc) | 86.576 | 87.233 |
| Grain Volume Vg (cc) | 72.209 | 72.088 |
| Grain Density (g/cc) | 2.591 | 2.645 |
| Permeability of air (mD) | 57.790 | 37.988 |
| Absolute permeability (mD) | 52.381 | 33.898 |

2. Properties of fluid

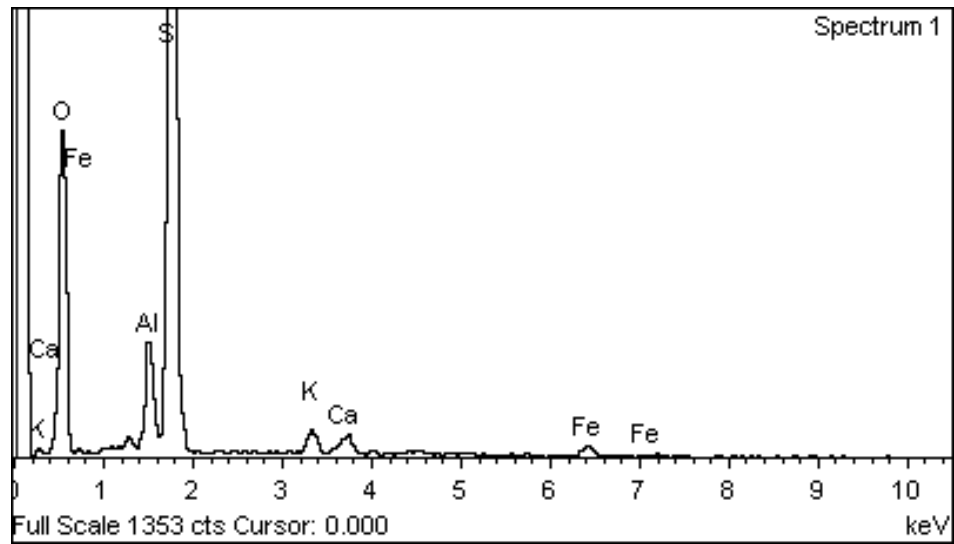
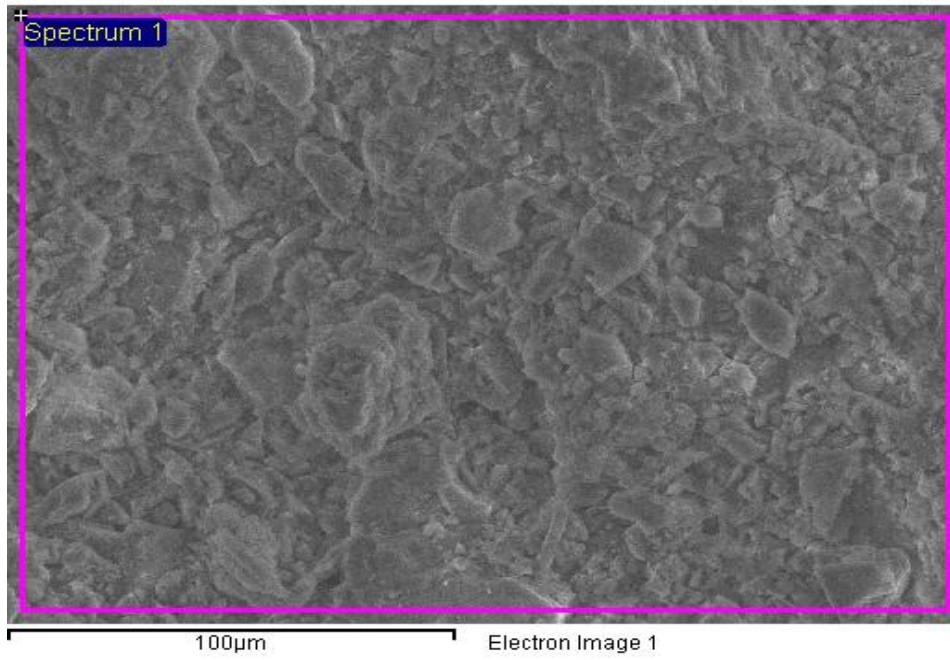
| | |
|--|--------|
| Viscosity of Dulang Crude oil @ 98 °C (cp) | 0.8000 |
| Density of Dulang Crude oil @ 98 °C (g/cm ³) | 0.5200 |
| Dulang Crude oil acid number (mg KOH/ g oil) | 0.9300 |
| Density of 1% NaCl (g/cm ³) | 0.9137 |
| Density of 0.5% NaOH (g/cm ³) | 1.0047 |
| Density of 1.5% NaOH (g/cm ³) | 1.0146 |

3. Core flooding displacement run result

| CORE 1 | |
|--------------------------------|-------|
| Oil Saturate | |
| Original Water Saturation (cc) | 14.37 |
| Displaced Brine (cc) | 12.80 |
| OOIP (cc) | 12.80 |
| Residual Water (cc) | 1.57 |
| Oil Saturation, So | 0.89 |
| Critical Water Saturation, Swc | 0.11 |
| Water Flooding | |
| Displaced Oil (cc) | 6.20 |
| Residual Oil (cc) | 6.60 |
| Water Saturation (cc) | 7.77 |

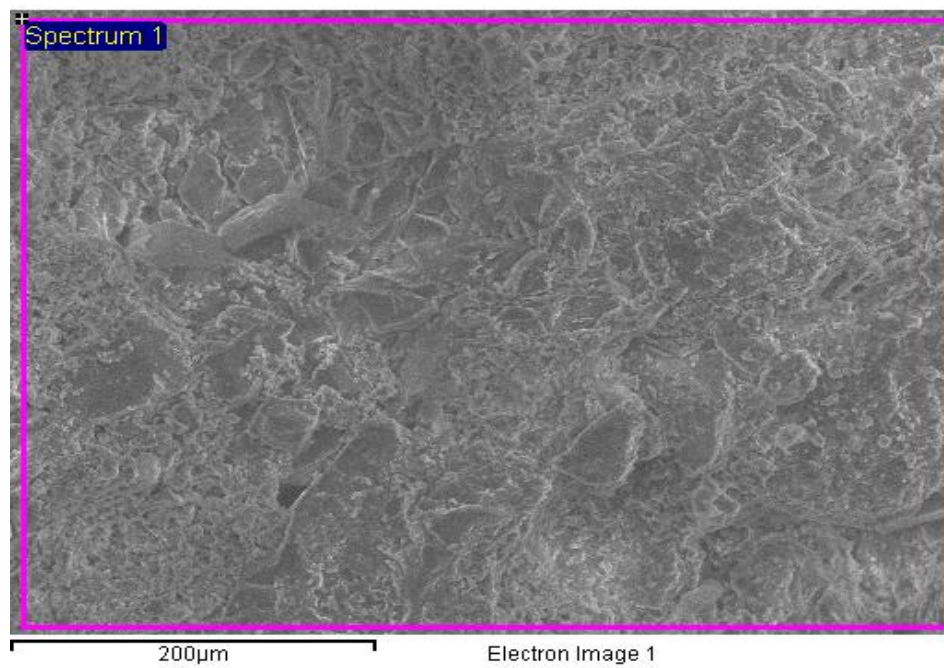
| | |
|-----------------------------------|-------|
| Critical Oil Saturation, Sor | 0.46 |
| Water Saturation, Sw | 0.54 |
| Alkaline Flooding | |
| Oil in place before alkaline (cc) | 6.60 |
| Additional Oil displacement (cc) | 3.50 |
| Residual Oil (cc) | 3.10 |
| Critical Oil Saturation, Sor | 0.22 |
| CORE 2 | |
| Oil Saturate | |
| Original Water Saturation (cc) | 15.15 |
| Displaced Brine (cc) | 9.00 |
| OOIP (cc) | 9.00 |
| Residual Water (cc) | 6.15 |
| Oil Saturation, So | 0.59 |
| Critical Water Saturation, Swc | 0.41 |
| Water Flooding | |
| Displaced Oil (cc) | 6.00 |
| Residual Oil (cc) | 3.00 |
| Water Saturation (cc) | 12.15 |
| Critical Oil Saturation, Sor | 0.20 |
| Water Saturation, Sw | 0.80 |
| Alkaline Flooding | |
| Oil in place before alkaline (cc) | 3.00 |
| Additional Oil displacement (cc) | 2.50 |
| Residual Oil (cc) | 0.50 |
| Critical Oil Saturation, Sor | 0.03 |

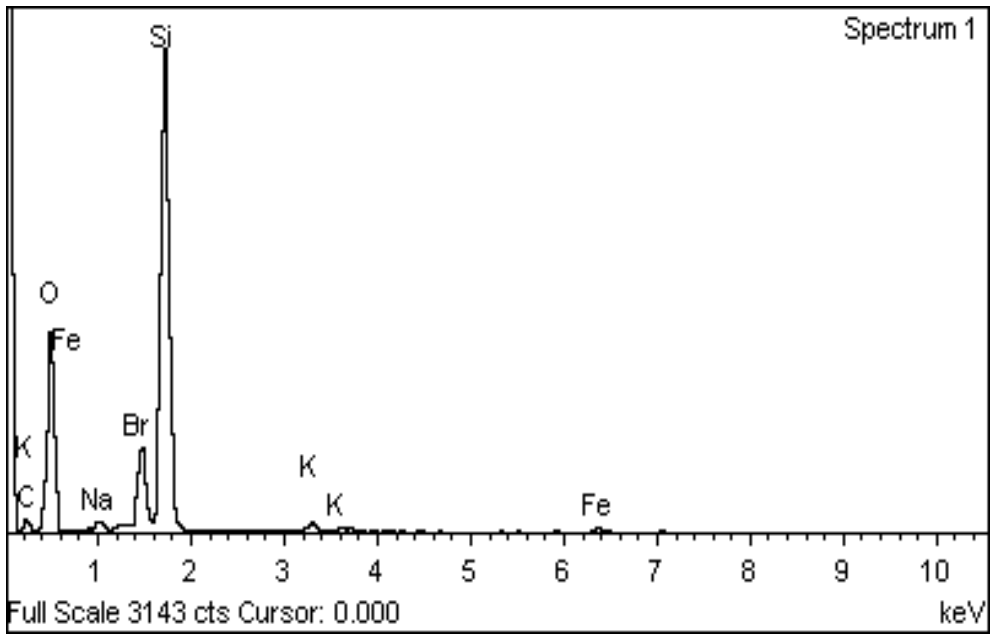
4. EDX of Berea sandstone sample



| Element | Weight% | Atomic% |
|---------|---------|---------|
| O K | 54.63 | 68.66 |
| Al K | 4.18 | 3.11 |
| Si K | 36.42 | 26.08 |
| K K | 1.66 | 0.85 |
| Ca K | 1.24 | 0.62 |
| Fe K | 1.88 | 0.68 |
| Totals | 100.00 | |

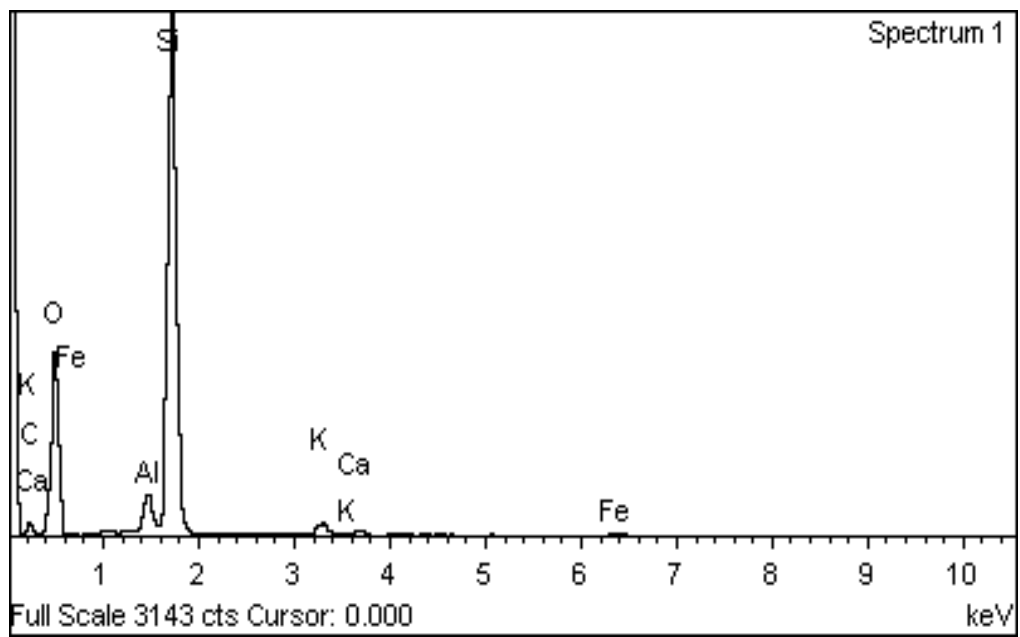
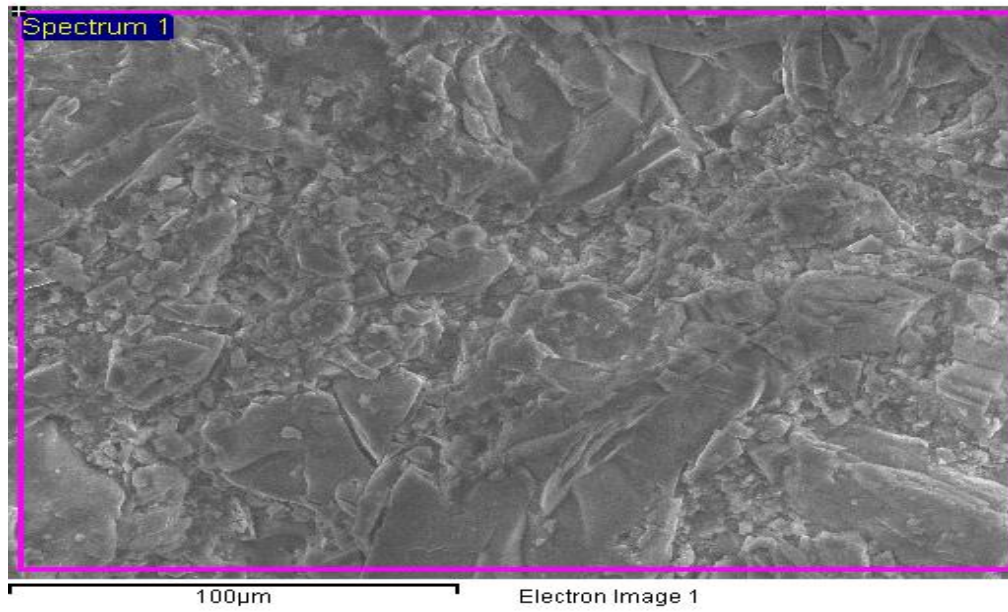
5. EDX of core 1 after core flood





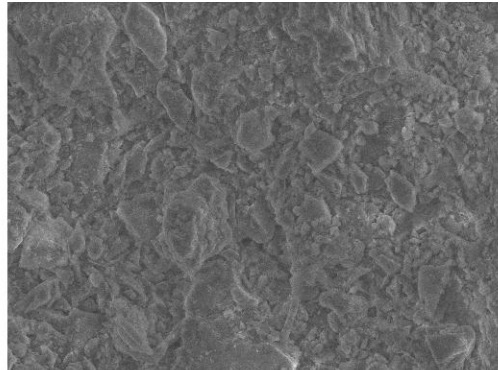
| Element | Weight% | Atomic% |
|---------|---------|---------|
| C K | 16.42 | 25.23 |
| O K | 47.61 | 54.92 |
| Na K | 0.66 | 0.53 |
| Si K | 25.55 | 16.79 |
| K K | 0.67 | 0.32 |
| Fe K | 1.23 | 0.41 |
| Br L | 7.85 | 1.81 |
| Totals | 100.00 | |

6. EDX of core 2 after core flood

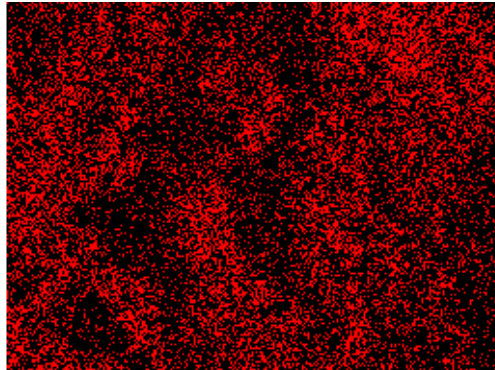


| Element | Weight% | Atomic% |
|---------|---------|---------|
| C K | -4.62 | -7.95 |
| O K | 57.35 | 74.04 |
| Al K | 2.54 | 1.94 |
| Si K | 41.20 | 30.30 |
| K K | 1.60 | 0.85 |
| Ca K | 0.68 | 0.35 |
| Fe K | 1.26 | 0.47 |
| Totals | 100.00 | |

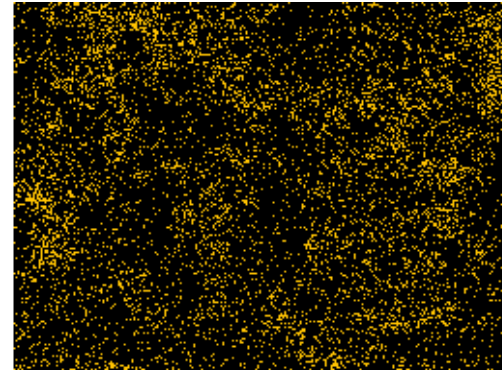
7. Minerals mapping for Berea sandstone sample



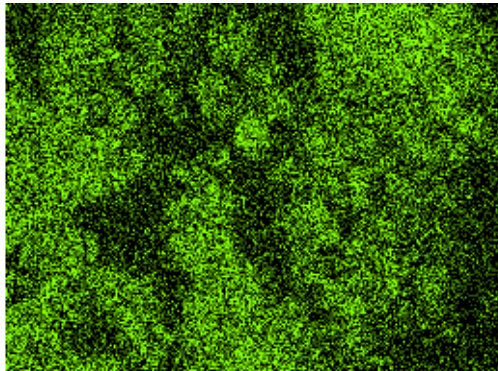
Electron Image 1



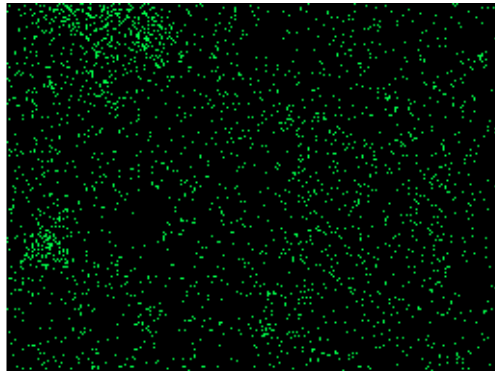
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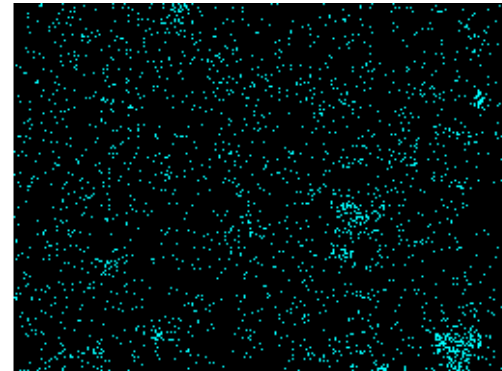
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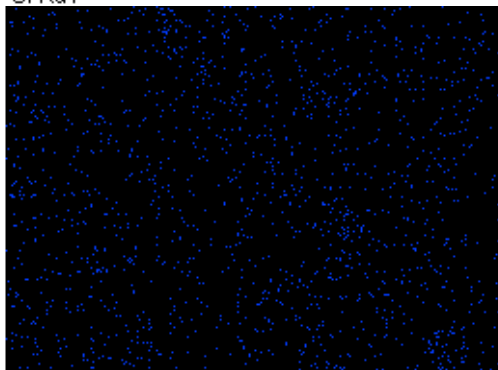
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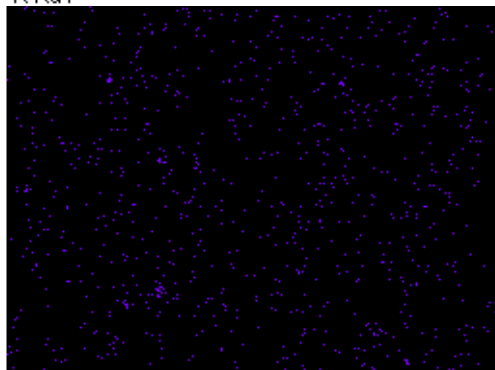
K Ka1



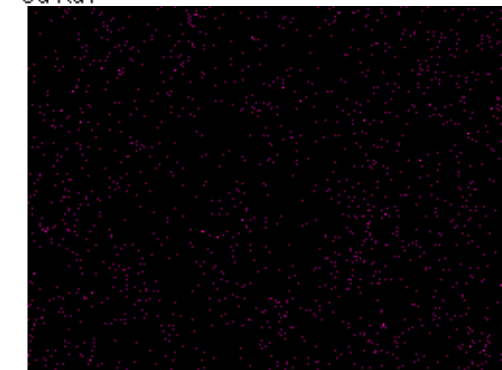
Ca Ka1



Fe Ka1

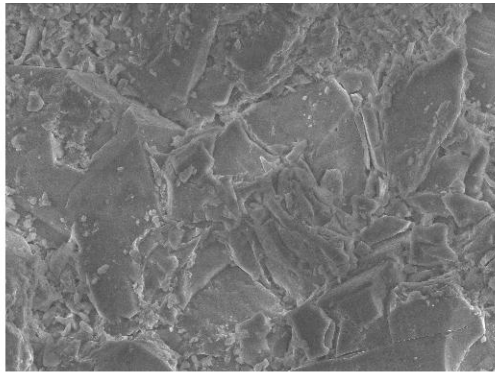


Ti Ka1

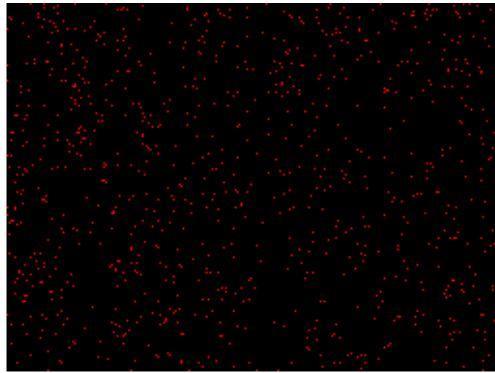


Mg Ka1_2

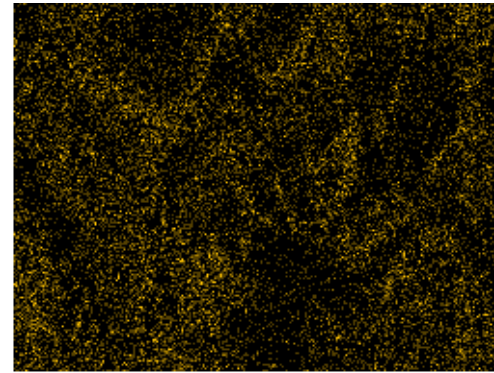
8. Minerals mapping from core 1 after core flood



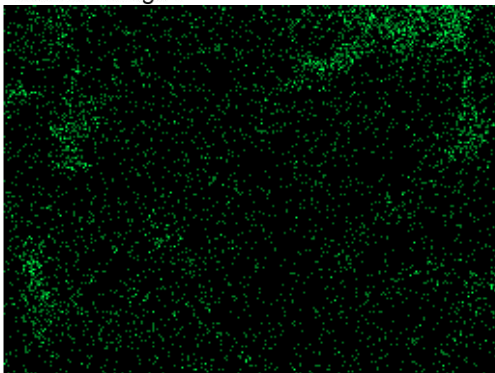
Electron Image 1



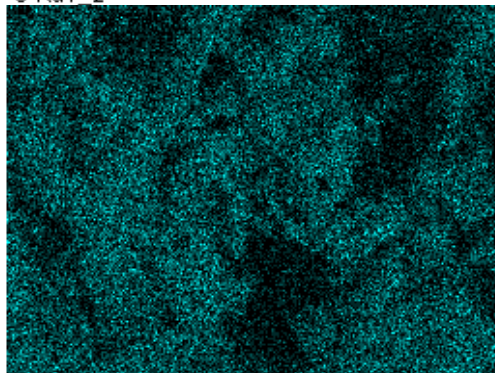
C Ka1 2



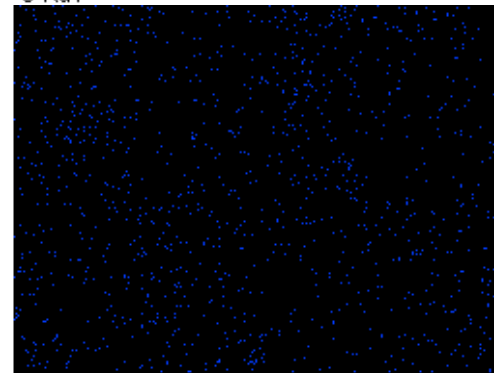
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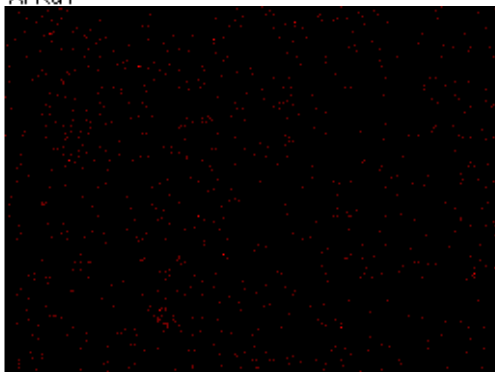
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Si Ka1

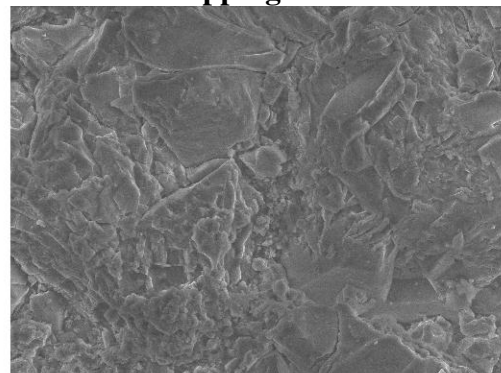


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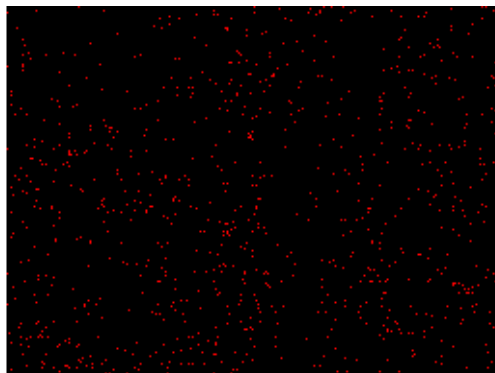


Fe Ka1

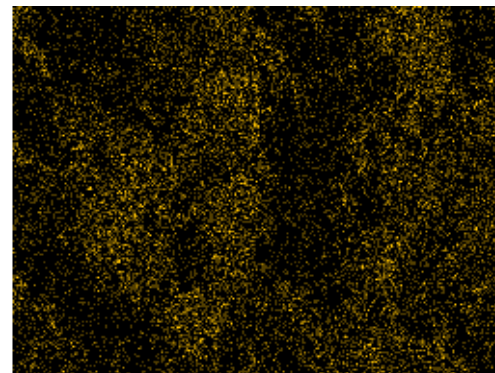
9. Minerals mapping for core 2 after core flood



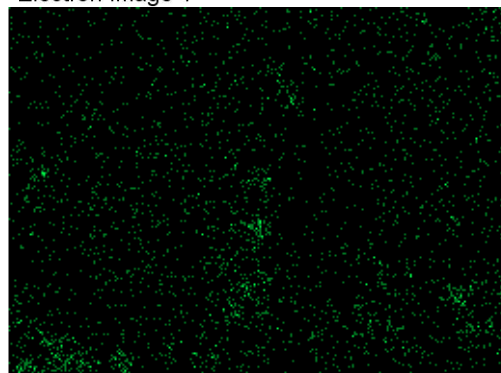
Electron Image 1



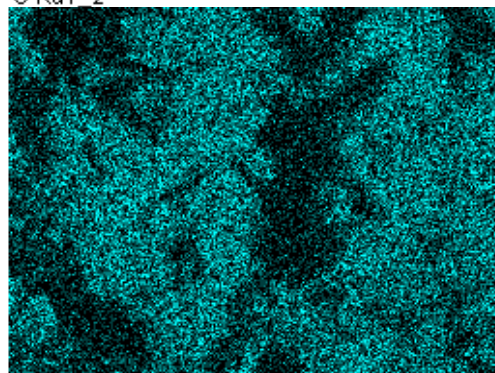
C Ka1 2



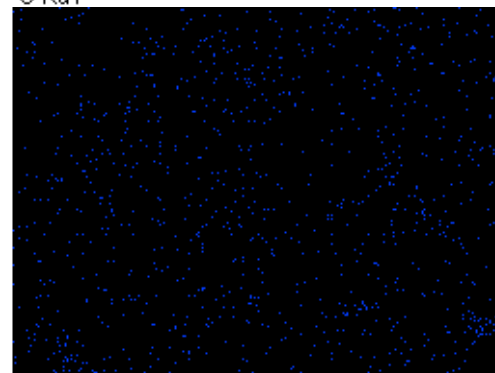
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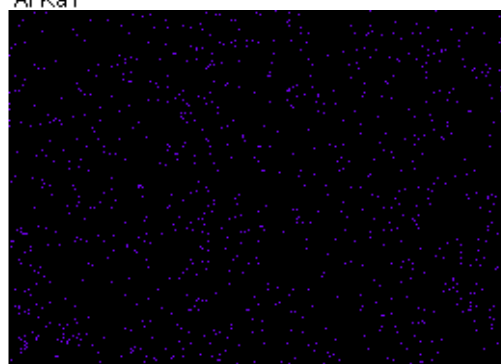
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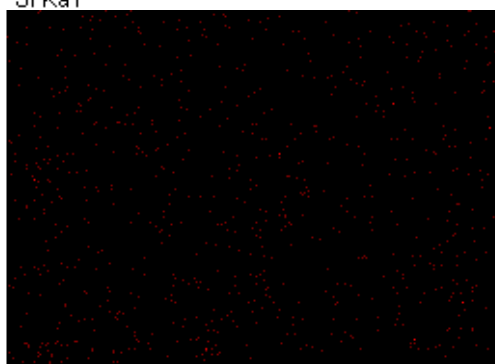
Si Ka1



K Ka1



Ca Ka1



Fe Ka1