CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Porous Media

- Sand pack samples: Ottawa sand, U.S. Silica Company

3.1.2 Fluids and Chemicals

- Deionized water with density of 1 g/ml and viscosity of 1 cP at room temperature (~ 25°C)
- Fluorolube oil, $[CF_2\text{-}CFCl]_n$: FS-5 Grade, Gabriel Performance Products
- Conformance Control Polymer: Alcoflood 935, Ciba Specialty Chemicals
- Oxygen scavenger: Na_2SO_3, Sigma Chemical, Co.
- Crosslinker: Cr(III) acetate, McGean-Rohco, Inc.
- AquaSil™ Siliconizing Fluid, Pierce Biotechnology, Inc.

3.2 Equipment

Table 3.1 Instruments and models used in this work

Instrument	Model	Usage
Shaker	RX-29 model, W.S. Tyler Incorporated Ro-Tap®	Sand grain size analysis
Viscometer	Brookfield LVF model, 4 Speeds (60, 30, 12, and 6 RPM)	Fluid viscosity determination
Pycnometer	Volume of 10 ml	Fluid density determination
Infusion / Withdrawal Pump	model 944, Hardvard Apparatus, Holliston MA	Flood testing
ReciPro Pump	HP series model A-30-S, Eldex Laboratories, Inc.	Sample preparation
MRI Machine	7T/60/AS, Magnex Scientific Ltd., Oxford, UK	Sample visualization

3.3 Methodology

3.3.1 Analysis of Ottawa Sand Properties

Ottawa sand was used to represent the unconsolidated, high permeability porous media. The sand was sieved using a Shaker, RX-29 model, W.S. Tyler Incorporated Ro-Tap® to determine the grain size distribution. The effect of wettability on oil recovery and displacement characteristic was studied. The wettability of the sand was altered using AquaSil™ solution.

3.3.1.1 Sand Grain Size Analysis

a. Procedure:
   1. Weigh the total amount of sand.
   2. Sieving the grain size of the sand by running the sand through a specific sized screen for an hour. In this experiment, sieve series were used in the order of 20, 30, 40, 50, 60, 80, 100, 140, 200 mesh, respectively. Note that as the mesh number increases, the grain size passed through by that mesh decreases.
   3. Weigh the sand in each sized screen.
b. Calculations and report:
   1. Calculate the average size of sand by the weight of the sand in each screen. This means that the particles passed in each screen were trapped by the next smaller sized screen.
   2. Report the grain size distribution by weight.

3.3.1.2 Silica Sand Wettability Alteration

a. Procedure:
   1. Dry silica sand in an oven at 100°C for at least 1 hour.
   2. Prepare a diluted solution of AquaSil™ at 0.1 % with water (this solution should be prepared fresh daily). AquaSil™ contains 20 % active ingredient; therefore, a 1:100 dilution gives a 0.2 % solution. If the concentrate is hazy, filter to remove polymers. It may be necessary to make a more concentrated working solution if the concentrate becomes too polymerized. Stability can be enhanced by adjusting the aqueous solution to pH 4.5-5.0

using HCl. (AquaSil™ and SurfaSil™ Siliconizing Fluids data sheet, 2003)

3. Soak the sand with the diluted solution for at least 2 days. Agitate the solution to ensure a uniform coat. A thin film coats the surface of the sand making it water repellent.
4. Dry sand in an oven at 100°C for 1 hour to cure the silicone coating (Romero-Zerón, 2004).

b. Calculations and report:

1. Check the wettability behavior of the coated and uncoated silica sand by placing drop of colored water on a layer of sand in a glass container.
2. In the case of uncoated sand (water-wet sand), colored water rapidly imbibed the sand. In contrast (oil-wet sand), the water did not spread on the coated sand immediately and formed like a drop on the sand surface.

3.3.2 Analysis of Fluid Properties

Density and viscosity are the basic properties of the fluids which are important for displacement processes. Viscosities of displaced and displacing fluids describe the main parameter of the displacement process which is the mobility ratio. The polymer characteristics are also determined by viscosity that varies with the shear rate.

3.3.2.1 Fluid Density Determination by Pycnometer

a. Procedure:

1. Weigh the empty dry pycnometer (a flask with a close-fitting ground glass stopper with a fine hole through it, so a given volume can be accurately obtained).
2. Fill the pycnometer with the liquid sample. Weigh the pycnometer containing the liquid.

b. Calculations and report

1. Take the different between the mass of pycnometer containing the liquid and the mass of the empty pycnometer to get the mass of the liquid sample.

2. Calculate the density of the liquid sample (ρ) by knowing the mass of the liquid sample (M) and the volume of the pycnometer (V), the density of the liquid sample can be obtained using the equation $\rho = M/V$.

3.3.2.2 Fluid Viscosity Determination by Viscometer

a. Procedure (Synchro-lectric Viscometer LVF Model, Instruction Manual):
   1. Attach the spindle to the lower shaft. Care should be taken to avoid putting side thrust on the shaft to protect its alignment.
   2. Insert the spindle in the liquid sample until the liquid level is at the immersion groove cut in the spindle shaft. With a disc type spindle, it is sometimes necessary to tilt the instrument slightly while immersing to avoid trapping air bubbles on its surface. Care should be taken not to hit the spindle against the sides of the liquid container.
   3. Level the viscometer. The bubble level will be of help in this respect.
   4. Check the pointer at zero, depress the clutch and turn on the viscometer motor, and then release the clutch and allow the dial to rotate until the pointer stabilizes at a fixed position on the dial. The time required for stabilization will depend on the speed at which the spindle rotates: at speeds above 4 RPM this will generally be about 20-30 seconds.
   5. Depress the clutch and snap the motor switch to stop the instrument with the pointer view.
   6. Record dial readings for each speed. Note that the minimum range is obtained by using the largest spindle at the highest speed – the maximum range by using the smallest spindle at the slowest speed.

b. Calculations and report:
   1. Calculate the viscosity in centipoises (cP) of the liquid by multiplying the dial reading using the factor in Table 3.2.

Table 3.2 Factor for viscosity calculation (Synchro-lectric Viscometer LVF Model, Instruction Manual)

Spindle number	RPM	Factor
1	60	x1
	30	x2
	12	x5
	6	x10
2	60	x5
	30	x10
	12	x25
	6	x50

Spindle number	RPM	Factor
3	60	x20
	30	x40
	12	x100
	6	x200
4	60	x100
	30	x200
	12	x500
	6	x1000

2. Report the average viscosity for a Newtonian fluid, its viscosity is constant. In this experiment Newtonian fluids are water and Fluorolube FS-5.
3. Plot a relationship of viscosity and shear rate (corresponding to the speed, RPM) for non-Newtonian fluids, its viscosity varies with changing rate of shear. In this experiment the non-Newtonian fluid is a polymer.

3.3.3 Polymer Preparation

Alcoflood 935, which is a partially hydrolyzed polyacrylamide (HPAM), is the polymer used in this study. This anionic acrylamide copolymer has been supplied by Ciba Specialty Canada Inc., which classifies Alcoflood 935 as a conformance control polymer. Alcoflood 935 has a degree of hydrolysis of about 10% and manufacturer-reported molecular mass of $8x10^6$ – $10x10^6$ g/mol. Three polymer solutions were prepared dissolving 0.25 wt%, 0.5 wt%, and 0.75 wt% of HPAM. An oxygen scavenger, Na_2SO_3, was used at a concentration of 0.01 wt% to prevent the degradation of the polymer chains (Wassmuth *et al.*, 2001).

a. Procedure:
   1. Weigh the water and polymer, Alcoflood 935 (powder form) in the ratio of 99.25:0.75 for 0.75 wt% concentration, 99.50:0.50 for 0.50 wt% concentration, and 99.75:0.25 for 0.25 wt% concentration.
   2. Put Na_2SO_3 (oxygen scavenger) in the ratio of 0.01 g per 100 g of solution to prevent polymer degradation.
   3. Shake the solution overnight with a speed of 640-720 RPM.

3.3.4 Polymer-Gel Preparation

Cr(III) acetate solution, 50% active in water (pH=2.6), supplied by McGean-Rohco, Inc. is the crosslinker used in this study. 0.3 wt% of Cr(III) acetate in 0.75 wt% HPAM was prepared as a polymer gel.

a. Procedure:
   1. Keep 0.75 wt% of polymer solution at temperature of 40 °C.
   2. Weigh the polymer solution and Cr(III) acetate in the ratio of 99.7: 0.3
   3. Mix the solutions immediately.
   4. Keep the polymer-gel solution at a temperature of 40°C.

3.3.5 Analysis of Porous Media Properties

For waterflooding and polymer flooding processes in water-wet sand and oil-wet sand, the sand pack sample was packed in the syringe that has a volume of 44.07 cm^3 (2.6 cm in diameter and 8.3 cm in length) as shown in Figure 3.1.

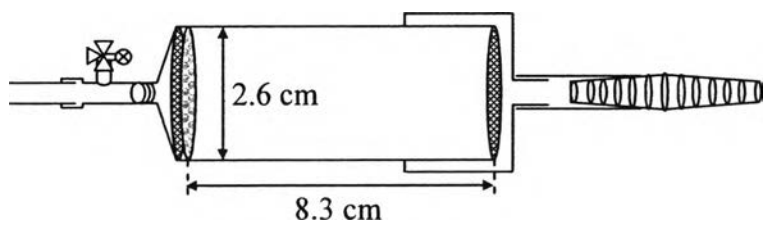

Figure 3.1 Tube dimensions of the sand pack sample

The sample was saturated with water at a reservoir flow rate (Frontal Advanced Rate, FAR of 2 ft/day which is approximately 0.06 ml/min) to measure the

pore volume, porosity and absolute permeability to water. Then, the sample was saturated with Fluorolube FS-5 at the reservoir flow rate and followed by a high flow rate to correct the end effect until residual or irreducible water saturation (S_{wirr}) was reached (Huang and Honarpour, 1998). A ReciPro Pump was used to drive the water in this section as shown in Figure 3.2. The sample was then visualized during the core flood test by MRI. A schematic diagram of the overall experiment is shown in Figure 3.3.

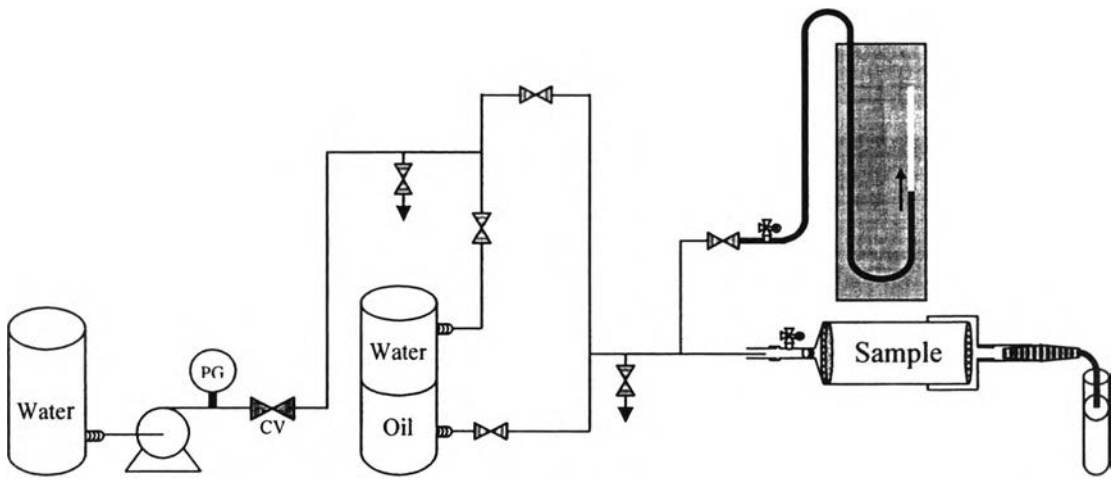

Figure 3.2 Schematic of the experimental setup for sample preparation

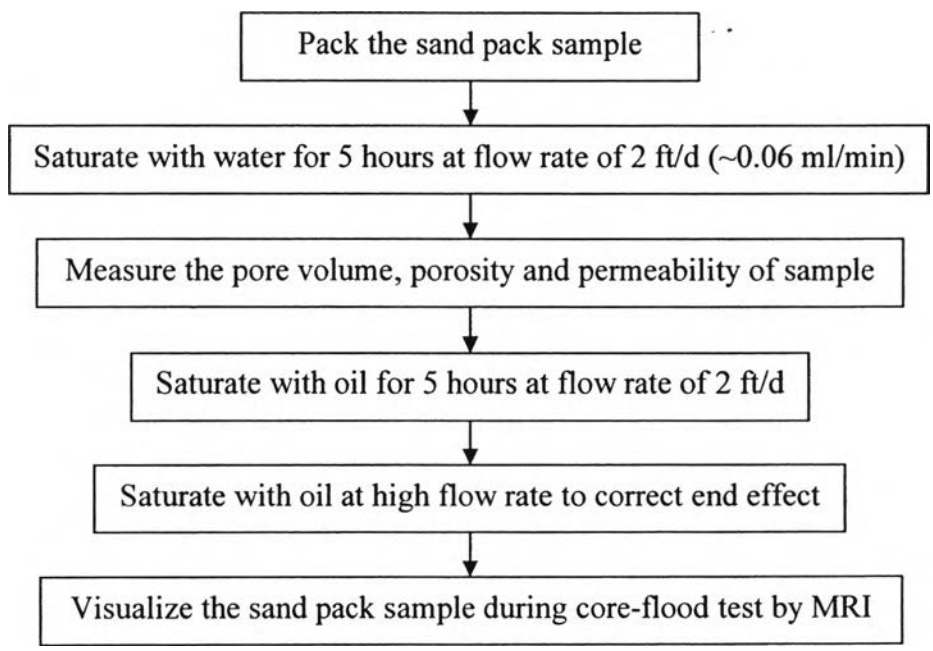

Figure 3.3 Schematic diagram of overall experiment

For the channel system, a syringe of 37.17 cm^3 (2.6 cm in diameter and 7 cm in length) was used as the cylindrical cell for sand pack. The strongly water-wet glass tube was placed in the center of the sand pack to represent the high permeability channel. A fluid diffuser plate at the inlet-face of the sand pack was connected with the channel. The channel has a volume of 0.4 cm^3 which had an inside and outside diameter of 0.23 and 0.41 cm, respectively and a length of 9.5 cm. Figure 3.4 shows the channel model with all dimensions as described.

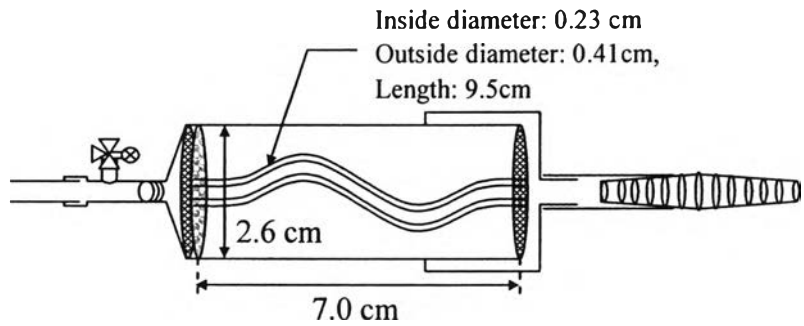

Figure 3.4 Tube dimensions for the channel system

The sample was saturated with water at the reservoir flow rate to determine the pore volume, porosity, and permeability in the sand matrix. The procedure details for each sample will be explained later in the Chapter 4. The subsequent step in the preparation of the sand pack was the drainage of the unconsolidated porous media using Flourolube FS-5 at the reservoir flow rate, followed by a high flow rate to correct end effects until irreducible water saturation (S_{wirr}) was reached. At this point, the sample was ready to visualize the displacement process by MRI.

3.3.5.1 Porosity Determination by the Liquid Saturating Method

a. Procedure:
   1. Weigh sample container, $W_{container}$, measure its diameter D, and length L in order to calculate container volume, V_b.
   2. Weigh dry sand sample, W_{dry}.
   3. Saturate the sample with distilled water, $\rho_{water} = 1$ g/cm^3.
   4. Weigh the saturated sample, W_{sat}.

b. Calculations and report:
   1. Calculate the saturated distilled water weight, $W_{water} = W_{sat} - W_{dry} - W_{container}$.
   2. Calculate the pore volume (saturated distilled water volume), $V_p = W_{water} / \rho_{water}$.
   3. Calculate the porosity, $\phi = V_p/V_b$.

3.3.5.2 Absolute Permeability Measurement of Water

a. Procedure:
   1. Measure Length L, and diameter D of sample container in order to calculate container cross section area A.
   2. Measure the pressure drop ΔP under operational flow rate. Each measurement collects water production V_w, in time ΔT.

b. Calculations and report:
   1. Calculate the flow rate of water produced, $Q_w = V_w/\Delta T$.
   2. Plot a relationship of Q_w/A and $\Delta P/L$ to obtain the slope of K/μ.

3.3.6 Visualization of Porous Media during Core-Flood Test by MRI

The sand pack sample was placed in the MRI machine during the core-flood test. A tube sample of Ottawa sand saturated with Fluorolube FS-5 was placed as a reference above the sand pack sample during MRI visualization as shown in Figure 3.5. An Infusion/Withdrawal pump was used to drive the water or polymer with varying concentrations of 0.25 wt%, 0.50 wt% and 0.75 wt% in order to flood the sample with a certain flow rate until residual oil saturation (S_{or}) was reached.

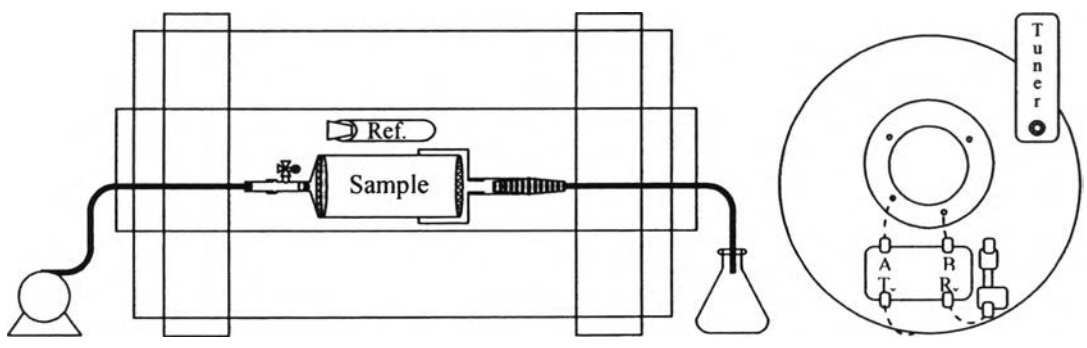

Figure 3.5 Schematic of the MRI experimental setup

3.3.6.1 MRI Machine Specification

The MRI Machine was performed on a MARAN spectrometer (Resonance Instrument Ltd., Oxford, UK) with a 7 Tesla, widebore, horizontal superconducting magnet 7T/60/AS (Magnex Scientific Ltd., Oxford, UK). The standard micro-imaging gradient set SGRAD156/100/S (Magnex Scientific Ltd., Oxford, UK) employed was powered by a set of three gradient amplifiers 7782 (AE Techron, Elkhart, USA), providing a maximum gradient strength of 38 G/cm. A home-made 62 mm inner diameter RF probe was used with an RF power amplifier 7T100S (Communication Power Corp., New York, USA). All measurements were carried out at 15°C inside the probe.

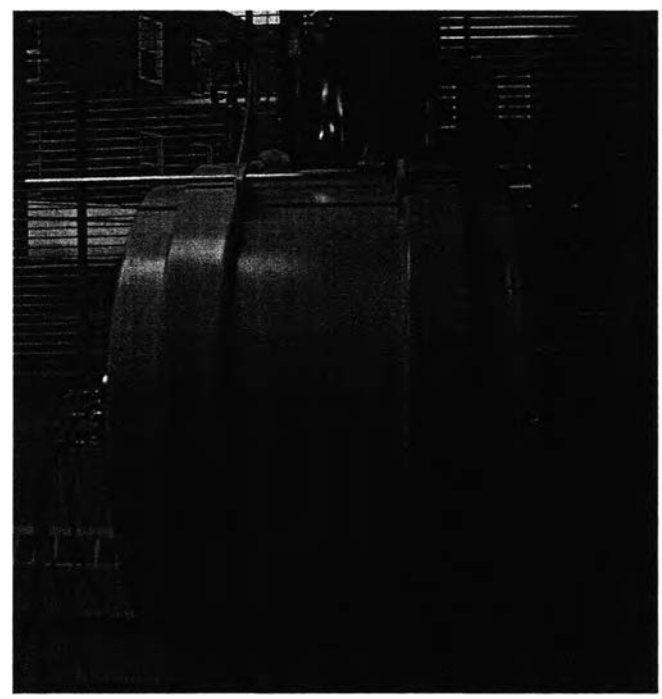

Figure 3.6 Photograph of MRI machine

3.3.6.2 MRI Parameters and Data Processing

Acquisition parameters for conical SPRITE 3D-measurement were: Matrix 64x64x64, interleaves = 39, field of vision (FOV) = 100x100x100 mm, encoding time (t_p) = 70 μs, repetition time (TR) = 500 μs, Flip angle = 2°. The delay between the gradient interleaves was 500 ms. Acquisition time = 1.5 min, Number of scan = 4.

Multiple FID points acquired = 5 points, dwell time (*DW*) = 5 μs. In this work, the centric scan SPRITE MRI technique was selectively conducted for ^{19}F to visualize fluorinated oil such as Fluorolube oil in the porous media.

The Acciss, Unifit and Impstar processing packages developed in the IDL programming environment by the UNB MRI Research Centre were used for image reconstruction, image processing and image display. The WinDXP program was used for relaxation time distribution fitting.

The oil saturation during the flooding experiment was tracked by an integral total MRI signal from the sample. A material balance was applied to validate the MRI measurement by determination of residual oil saturation and oil recovery after the flooding processes.