CHAPTER III
EXPERIMENTAL SECTION

3. 1 Batch Experiment

3. 1. 1 Preparation of Silica Particles in Microemulsions

The highly purified nonionic surfactant Igepal RC520 (DP6) was kindly supplied by Rhone-Poulenc Inc. and used to prepare water in oil (W/O) microemulsion solutions. This surfactant is a polydisperse mixture of poly(oxyethylene) alkylphenylether molecule with the structure n-$H(CH_2)_{12}Ph$-$(OC_2H_4)_6OH$. Tetraethyl orthosilicate (TEOS)($Si(OC_2H_5)_4$) with 99.999% purity (Aldrich), aqueous ammonia consisting of 29.6 wt % ammonia (NH_3), and the co-surfactants of 1-butanol, 1-octanol, and 1-dodecanol were used without further purification. HPLC grade heptane was used as a carrier fluid to prepare W/O microemulsion solution. The W/O microemulsions were prepared by addition of aqueous ammonia and purified water to the heptane solution containing premixed DP6 and one type of co-surfactant. Then, the resulting mixture was gently shaken till completely transparent. The hydrolysis of TEOS and the formation of silica particles occurred immediately after addition of TEOS into microemulsions. The entire reaction was carried out at a temperature of 22^0 C in the 20 ml vials under different conditions.

3. 1. 2 Measurement and Analysis Methods

3.1.2.1 UV-Visible Spectrophotometric Measurement

The relative particle density was measured by UV-Visible spectrophotometer (Varian Model Cary 100 Bio). The absorbance of silica particles was measured at the wavelength 500 nm. Due to the various possible sizes of the silica particles produced in each reactor, the absorbance value can only give a relative degree of turbidity.

3.1.2.2 Fourier Transform Infrared Spectroscopic Measurement

Fourier-transform infrared spectroscopy (FTIR) was used to study the kinetics of TEOS hydrolysis in microemulsion. The concentration of TEOS was quantified from the intensity of the Si-O-C streching band located at 967 cm^{-1}. The transmission FTIR spectra of microemulsion solutions were measured by using a single-beam Galaxy FTIR spectrophotometer. The microemulsion solution was accommodated with a pair of zinc-selenide (ZnSe) windows with Teflon Spacer 0.5 mm thick (Harrick Scienctific).

3.1.2.3 Transmission Electron Microscopic Measurement

The size and morphology of silica particles synthesized in microemulsions were investigated using a Jeol 2000 FX transmission electron microscope (TEM). The specimen for TEM analysis was prepared by placing a small amount of microemulsion solution on a 300-mesh carbon-coated copper grid (SPI Supplied). Dichloromethane was used to wash the residual surfactant remained on the copper grid off. The particle diameters, D, of silica particles were measured from the enlarged computer image of the TEM micrographs by using Image- Pro Express program.

3. 2 Linear Coreflood Experiment

A cylindrical ceramic core (1 inch diameter) with an initial permeability approximately 0.6 mD was used in linear coreflood experiment. This core was cut to approximately 12 cm for use in the system. The core was dried at 110° C and then loaded into a Hassler Cell (Figure 3.1) and radial overburden pressure of approximately 1000 psi was applied to the core in order to avoid seepage of flow out of the core radially. Heptane and microemulsion solutions were contained in stainless-steel accumulators attached to the inlet line. All fluids were driven by an FDS-210, high pressure, pulse free pump. Pressure transducers were placed at the inlet and, along the core to measure the pressure drop across the length of the core as a function of time.

Absolute permeability determined under heptane-saturated conditions was used as a reference point (K_0), and the initial plugging experiment was performed by injecting microemulsion solutions (reacting mixtures) directly into this heptane-saturated media. Pressure drops were measured over 2-in interior section of core to eliminate experimental anomalies due to the capillary end effects and face plugging.

The permeability was determined from the Darcy's equation as follows:

$$\frac{q}{A} = \frac{K}{\mu}\frac{\Delta P}{L} \qquad (3.1)$$

where q = flow rate, cm^3s^{-1}

A = cross sectional area, cm^2

L = length, cm

μ = viscosity, cP

ΔP = differential pressure, atm

K = permeability, D

There were multiple injections done in this experiment. After each shut in period (20 hours), the microemulsion solution containing silica particles was injected into the core at a low flow rate of 0.1 cc/min and the pressure drop along the core was measured until it reached steady state (pressure drop value was constant).

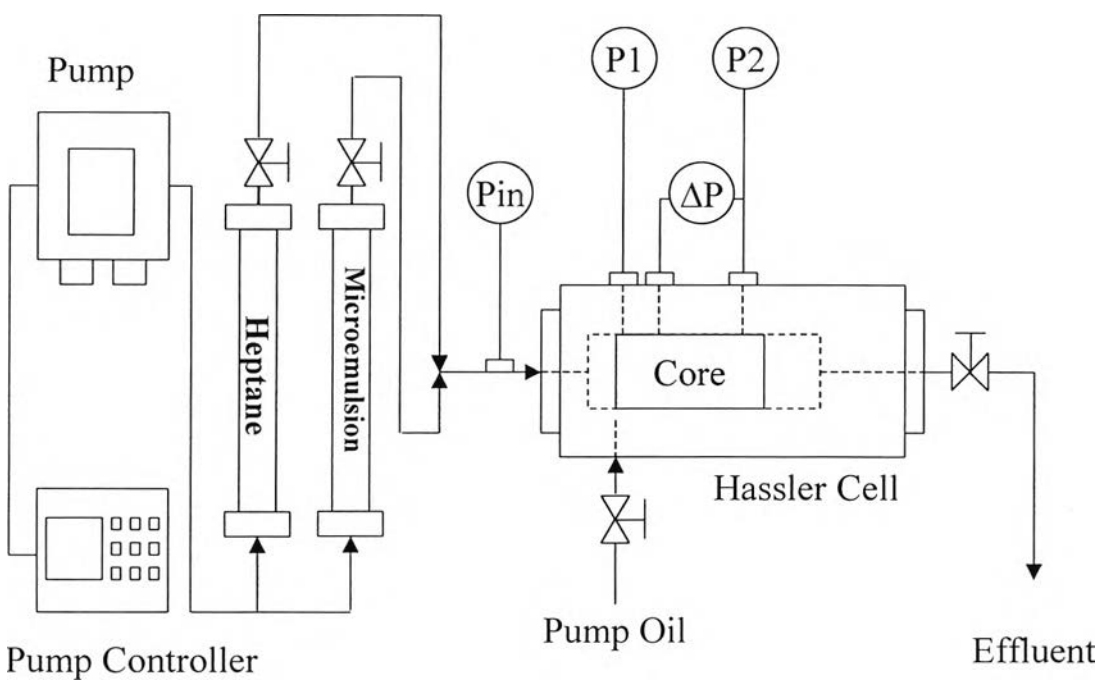

Figure 3.1 Schematic of coreflood apparatus.