CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Crude Oils

The crude oil, CH, used for experiments can be classified as medium/heavy oil because it has high asphaltene content but the viscosity is low compared to other high-asphaltene content crude oils. Another crude oil, GM3, is a light oil due to low asphaltene content and it has low viscosity also. The SARA composition and physical properties of CH crude oil are listed in Tables 3.1 and 3.2 respectively. The asphaltene content and physical properties in GM3 crude oil are shown in Table 3.3.

Table 3.1 SARA analysis of CH crude oil

Solubility Class	%wt in CH crude oil
Saturates	51.77
Aromatics	21.48
Resins	10.48
Heptane-Asphaltenes	10.56
Unrecovered fraction	5.71

Table 3.2 Physical properties of CH crude oil

Property	CH crude oil
Density at 20°C (g/mL)	0.8688
Viscosity at 60 °C (mPa*s)	8.93

Table 3.3 Asphaltene content and physical properties of GM3 crude oil

Property	GM3 crude oil
Heptane-Asphaltenes (%wt)	2.45
Density at 20°C (g/mL)	0.8680
Viscosity at 60°C (mPa*s)	4.65

3.1.2 n-Heptane Precipitant

N-Heptane was used to destabilize asphaltenes from the crude oil. Table 3.4 shows the properties and source of the n-Heptane used in this study.

Table 3.4 Physical properties at experimental condition (60°C) and source of precipitant

Precipitant	Density (g/ml)	Molar Volume (mL/mol)	Solubility Parameter ($MPa^{0.5}$)	Viscosity (mPa*s)	Purity	Source
n-Heptane	0.6494	154.3	15.2	0.272	99.4%	Fisher

3.1.3 Toluene

Toluene was used to clean the system and measure the diameter of capillary. Diameter measurement was done at 20°C. Table 3.5 shows the viscosity at the experimental conditions and source of toluene.

Table 3.5 Viscosity at experimental conditions and source of toluene

Solution	Viscosity at 20 °C (mPa*s)	Purity	Source
Toluene	0.585	99.9%	Fisher

3.2 Equipment

3.2.1 Optical Microscope Setup

An optical microscope (Nikon Eclipse E600) was used to detect the precipitation of asphaltene from crude oil-precipitant mixtures. The microscope setup provides a 50x total magnification. A monochrome Sony CCD video camera was connected with the microscope and linked to a Sony Camera Adaptor CMA-D2. The analog signal from the camera was converted to digital signal by a WinTV USB NTSC Model 40201 and digital images were captured at a resolution of 640 x 480 pixels.

3.2.2 Ultracentrifuge

Sorvall Legend X1R centrifuge was used for preparing crude oils.

3.2.3 Stainless Steel 316 Capillary Tubes

Stainless steel type 316 capillary tubes were used to detect deposition in the system. Two types were used in this experiment: precut capillary and capillary cut by rotary cutter.

3.2.4 Syringe Pumps

Teledyne ISCO Model 500D syringe pumps were connected to the cylinders as shown in Figure 3.1. Water was pumped from syringe into the cylinder to push the barrier inside the cylinder forward and solution is flowed to the system.

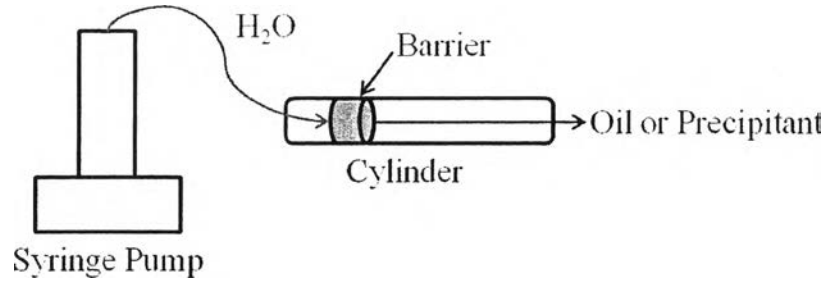

Figure 3.1 How solution was fed into the system using the syringe pump.

3.2.5 Water Bath

Cole-Parmer Polystat Heated Circulating Bath was used for controlling the environmental temperature, with an accuracy of ± 0.1°C.

3.2.6 Pressure Transducers

There are two pressure transducers in the system. First, the Sensotec A-5/882-15 was used to measure the pressure profile in the mixing section and the second one measured the capillary pressure drop by Sensotec Z/741-08ZD.

3.2.7 10 μm Stainless Steel Frit

There are two frits were used to improve the mixing and prevent asphaltene particle from mixing section. First, mixing frit, is put in mixing tee. For the second one, pre-filter, is located in connecting tee.

3.3 Methodology

3.3.1 Pretreatment of Crude Oil

In order to remove solid particles and water from crude oil samples obtained from the field, samples were centrifuged at 14,000 times of gravity force for 3 hours by a Sorvall Legend X1R centrifuge. Then, the particle-free crude oil was transferred from the top of the container (around 80% was collected) and kept in 250 mL reusable media/solution bottles with polypropylene plug-seal cap. The head space of the bottles was filled by nitrogen.

3.3.2 Preparation of Solution in Cylinder

Crude oil and precipitant (n-heptane) were carefully loaded into the sample cylinders to prevent air bubble as shown in Figure 3.2.

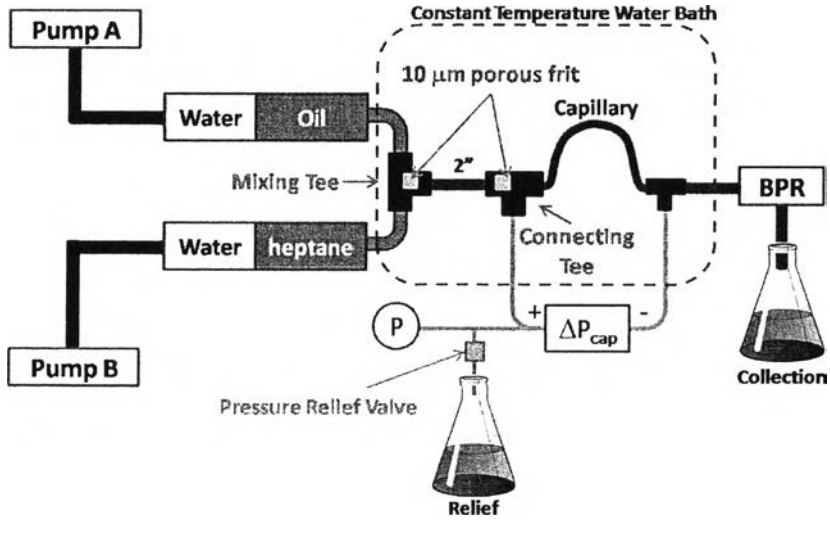

Figure 3.2 Asphaltene deposition apparatus.

3.3.3 Onset Experiment by Microscopy

To perform an onset experiment, the apparatus was connected without the pressure transducer as shown in Figure 3.2. Samples were taken from the effluent, shown as "collection" in Figure 3.2 by a micropipette and then dropped on a microscope slide. Micrographs were obtained at 50x total magnification. Crude oil was taken for the base case sample and then the desired concentration was changed by altering the oil and precipitant the flow rates. The effluent was collected and the micrographs were taken. The heptane concentration was increased until the onset point was reached.

3.3.4 Deposition Experiment

To begin a deposition experiment the mixing tee, connecting tee, capillary and effluent line were first connected without the outlet of the positive and negative pressure drop ($+\Delta P$ and $-\Delta P$) to the pressure transducer as shown in Figure 3.2. The precipitant line was partially filled with heptane, but an air pocket was left in the line before connecting the line to one side of mixing tee. The crude oil was prefilled into the system allowing it to move into the precipitant line but the air pocket prevents the mixing of the oil and precipitant. During the process of prefilling, oil will be maintained in the precipitant line by adjusting the flow rate of oil and precipitant before the Back Pressure Regulator (BPR) opened. Further, the positive and negative pressure drop outlets were connected respectively to the pressure transducer once the air has been forced out of the system. Finally, the desired flow rate of oil and precipitant were set. The system was placed in the water bath at $60^{\circ}C$. The data signal from the pressure transducer was collected by the program NI Datalogger.