

CHAPTER III EXPERIMENTAL

3.1 Materials

3.1.1 Crude Oil

Crude oil A was used in the all experiments and the SARA composition and physical properties are shown in Table 3.1.1.1 and 3.1.1.2 respectively.

Table 3.1.1.1 SARA composition of Oil A

Composition	Oil A (%wt)
Saturates	51.77
Aromatics	21.48
Resins	10.48
Asphaltene	10.56
Unrecovered fraction	5.71

Table 3.1.1.2 Physical properties of Oil A

Properties	Oil A
C7-Asphaltenes (%wt)	9.90 ± 0.07
Density at room temperature (20°C, kg/m ³)	869.2 ± 0.2
Viscosity at 60°C (mPa*s)	6.78 ± 0.07
Instantaneous onset at 60°C (vol. % heptane)	40%

3.1.2 Precipitants

n-hexane (C6), n-heptane (C7), n-octane (C8), n-nonane (C9), and n-decane (C10) were used as precipitants to destabilize asphaltene in Oil A. The physical properties of all precipitants in the experiments are shown in Table 3.1.2.1.

Table 3.1.2.1 Physical properties of Precipitants

Precipitant	Density at room temperature (20°C, kg/m ³)	Viscosity at 60°C (mPa*s)	Purity
n-hexane	660.6	0.212	HPLC Grade
n-heptane	679.6	0.276	HPLC Grade
n-octane	698.6	0.350	Reagent Grade
n-nonane	719.2	0.442	99%
n-decane	726.6	0.543	99%

3.1.3 Toluene

99% pure toluene was used to clean the system after finish the experiments.

The viscosity of toluene at 20°C is 0.585 mPa*s.

3.2 Equipment

3.2.1 Nikon Eclipse E600 optical microscope

A Nikon Eclipse E600 optical microscope was used to detect asphaltene particle in the solution. It was connected to a monochrome Sony CCS video camera which was linked to a Sony camera adaptor CMA-D2. It was then connected to a computer which has a WinTV USB NTSC Model 40201 program to capture images from the camera.

3.2.2 Sorvall Legend X1R Centrifuge

A Sorvall Legend X11R Centrifuge was used to pretreat Oil A by separating solid particles and water from Oil A.

3.2.3 Harvard apparatus 22 Syringe pump

A precipitant was added into Oil A at a constant flow rate using Harvard apparatus 22 Syringe pump in order to prepare the solutions.

3.2.4 Scale Calibration

In order to prepare the solutions to reach the desired precipitant concentration, the mass of solution was weighed using Scale Calibration and the concentration can then be calculated.

3.2.5 Eppendorf Micro Centrifuge 5418

The solution was transfer to 1.5 mL centrifuge tube and Eppendorf Micro centrifuge 5418 was used to separate asphaltenes from the solution.

3.2.6 Branson 1510 Sonicator

Branson 1510 Sonicator was used in the washing step in order to mix precipitant and asphaltene to wash the trapped Oil A in the asphaltenes.

3.2.7 Fisher Scientific Isotemp Incubator

Fisher Scientific Isotemp Incubator was used to evaporate precipitants trapped in the asphaltenes.

3.2.8 Sensotec A-5/882-15 pressure transducer

The pressure drop was measured using a Sensotec A-5/882-15 pressure transducer, a range of 10 psi, which was connected to a computer. A NI Datalogger program was used to record the pressure drop from transducer.

3.2.9 Cole-Parmer Polystat Heated Circulating Baths

Since all of the experiments were performed 60°C, Fisher Scientific Isotemp Incubator was used to control the temperature using water as a fluid medium.

3.2.10 Teledyne ISCO Model 500D Syringe pump

Teledyne ISCO Model 500D Syringe pumps were used to control flow rate of Oil A and precipitant during deposition experiments.

3.2.11 Stainless steel capillary

Stainless steel capillary, which is 0.01 inch in inner diameter and 5 cm in length, were used for deposition experiment purchasing from Upchurch Scientific with part number U-101 and Stainless steel capillary, which is 0.03 inch in inner diameter and 5 cm in length, were used in the mixing section purchasing from Upchurch Scientific with part number U-115.

3.3 Software

3.3.1 WinTV2000

3.3.2 NI Datalogger

3.3.3 MatLab

3.4 Methodology

3.4.1 Crude Oil Pre-Treatment

The Oil A used for experiments was first pretreated by centrifugation at 10,000 rpm for 3 hours to separate solid particles and water. The supernatant was then transferred to amber bottles for storage. In order to minimize the effect of oxidation, the storage bottle head space will be purged with nitrogen.

3.4.2 Centrifugation Experiment

Figure 3.4.2.1 shows a schematic diagram of centrifugation experiment. A precipitant was added to the crude oil to reach the desired precipitant concentration in order to destabilize asphaltenes and the samples were put in the water bath that was controlled the temperature to be 60°C. Samples were taken at various times (t_1 , t_2 , t_3 , etc.) and then transferred to two 1.5 mL microcentrifuge tubes. The microcentrifuge tubes were centrifuged at 14,000 rpm for 10 minutes to separate the precipitated asphaltenes. The supernatant was removed and additional precipitant was added to the asphaltene cake in order to wash off the trapped crude oil. The asphaltene cake was washed many times until the supernatant was a clear solution. After that, the asphaltene cake was dried in an oven and the weight of precipitated

asphaltene was measured. The sampling procedure was repeated until the weight of asphaltenes is constant.

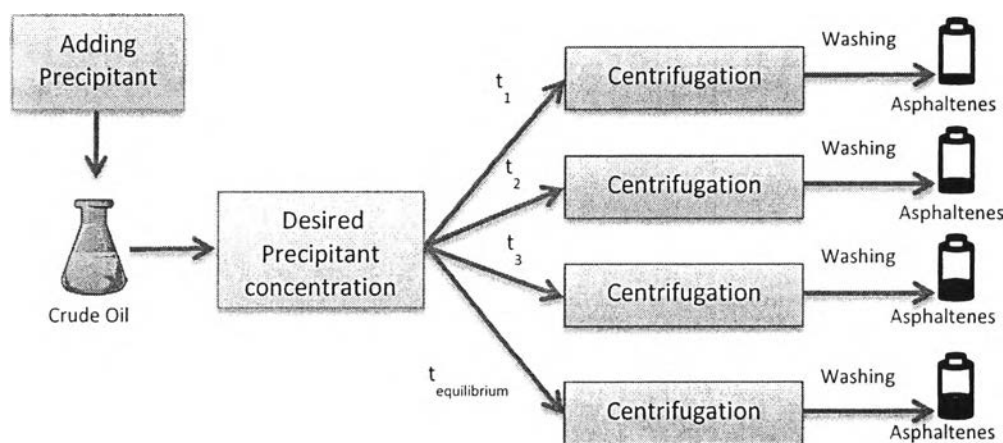


Figure 3.4.2.1 Schematic of a centrifugation experiment.

3.4.3 Onset Experiment

Precipitant was added into crude oil at a constant flow rate using a syringe pump to reach the desired precipitant concentration and samples were then put into a water bath that was controlled at the temperature 60°C . Samples were taken as a function of time and observed using optical microscopy. The time required for asphaltenes to reach the size of 0.5 micrometer was recorded as the detection time, $t_{\text{detection}}$.

3.4.4 Asphaltene deposition

Capillary flow was used to investigate the asphaltene deposition rate, and the apparatus used is shown in Figure 2.4.1. First, the precipitant was prefilled in the precipitant line in order to create air gab between the precipitant and a mixing tee. This air gap was created to prevent mixing of oil and heptane. The precipitant line was then connected to the mixing tee in the mixing section and Oil A was prefilled into the system resulting some oil flowing into the precipitant line. However, the air pocket prevented the precipitant and Oil A from mixing together. The flow rates of precipitant and Oil A were controlled using pumps 1 and 2 and the total flow rate for all experiment was 5 mL/h. After that, the positive and negative pressure drop lines

were respectively connected to the differential pressure transducer after the air had been bled out from the line. Finally, the flow rates were adjusted to achieve the desired precipitant concentration and the system was put in a water bath at 60°C. Precipitant and Oil A were then mixed inside a mixing section, and flowed through the capillary. Proper mixing in the system was obtained using porous frits in the mixing section and the pressure drop across the capillary was measured as a function of time.

The started time of the experiments is when a precipitant first enters the system, which can be observed from the decrease in the pressure drop due to the reduced viscosity with heptane in the mixture. In addition, the experimental pressure drop was first shifted using the average pressure drop, ΔP_0 , which is the average value of pressure drop before the pressure drop increases due to deposition. Moreover, Scanning Electron Microscopy (SEM) on a FEL Quanta 200 3D instrument operating at 0.45 torr in low vacuum mode was used to investigate the mixing in used capillaries.

3.4.5 Measuring diameter of capillary

The pressure drop across a capillary was used to measure diameter of capillary using DI water as a fluid medium. After the capillary was connected to the system, it was put into a water bath that was controlled at the temperature 25°C. The flow rate was adjusted to be 0.5, 1, 3, and 5 mL/min and the pressure drop was measured. The Hagan-Poiseuille equation, as shown in equation 2.4.1, was used to calculate the diameter of capillary.