

## CHAPTER III EXPERIMENTAL

### 3.1 Chemical and Equipment

#### 3.1.1 Chemicals

##### 3.1.1.1 *Crude Oil*

The crude oil sample was brought from Lankrabue field for this work called “new crude or LK2”. Lankrabue crude which was used in previous work (Numura, 2005) was also used for comparison purposes and called “previous crude or LK1”.

##### 3.1.1.2 *Polymer Chemicals*

- Poly(maleic anhydride-alt-1-octadecane or PMAO (99.9% purity)
- Poly(ethylene-co-butyl acrylate-co-maleic anhydride) or PEBAMA (99.9% purity)
- Poly(ethylene-co-vinyl acetate) or EVA with 18%, 25%, 33% and 40% vinyl acetate content
- Poly(lauryl methacrylate-co-ethylene glycol dimethacrylate) or PLEGMA

All above polymers were obtained from Aldrich Company.

- Poly(octadecyl methacrylate or POMA (99.9% purity) was obtained from Calro Erba.
- Poly (methyl methacrylate) or PMMA (VH and MD grades, 99.5% purity) were supplied by Siam Cement Co. Ltd.

Structures of these some polymers are shown in Figure 3.1.

##### 3.1.1.3 *Hydrocarbon Solvents*

- n-Pentane 99.5% purity
- n-Hexane 99.5% purity
- n-Heptane 99.5% purity

- Toluene, AR
- o-Xylene, AR
- Methanol, AR
- Acetone, AR
- Carbon disulphide 99.5% purity

All solvents was obtained from Labscan Company.

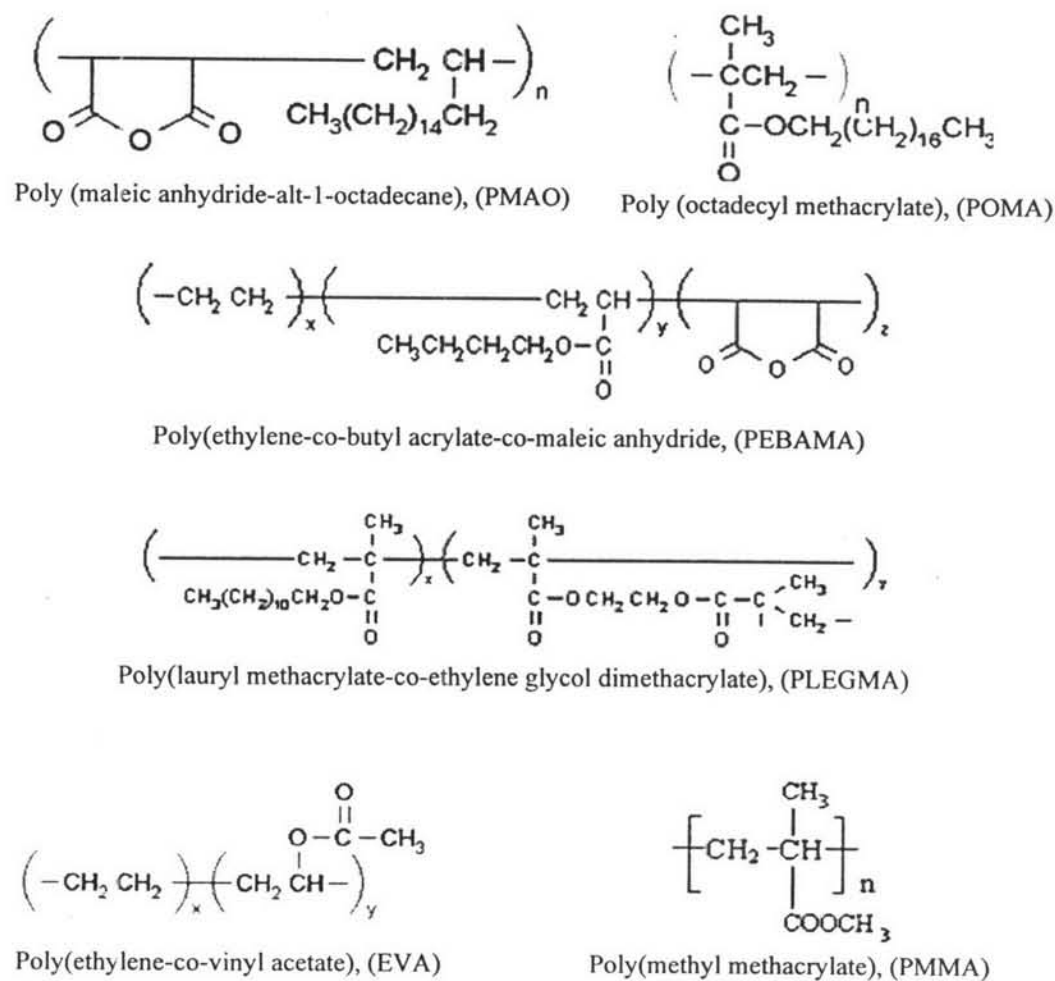


Figure 3.1 Molecular structure of polymers used in this study.

### 3.1.2 Equipment

- Stanhope-Seta pour point apparatus
- Simulated distillation gas chromatography (Sim-Dist GC), Varian GC-3800 model, Australia.
- Differential scanning calorimeter (DSC), Perkin Elmer 2800, USA.

## 3.2 Methodology

### 3.2.1 Sample Preparation

Normally, at room temperature crude oil samples are semi-solid. Crude oil in gallon plastic container was heated to 48°C by immersing the container in hot water for at least 1 hour in order to get the homogeneous crude. Then the same portions of homogeneous crude oil from each container were mixed and collected in a new container.

### 3.2.2 Crude Oil Characterizations

#### 3.2.2.1 *Crude Oil Composition*

A Varian GC-3800, model Australia, simulated distillation gas chromatograph (Sim-Dist GC) was used to identify hydrocarbon components by following ASTM D2887. The Sim-Dist GC was equipped with an on-column injector and a 10 m x 0.53 mm i.d., and 0.88 µm CP7512 capillary column with flame ionization detector (FID). Helium was used as a carrier gas at the flow rate of 60 ml/min. Following ASTM D2887, the column was programmed from 30°C to 380°C at the program rate of 10 °C/min. The crude oil sample was heated to 60°C to get the homogenous crude and dissolved in carbon disulfide (CS<sub>2</sub>) and the concentration of 0.01 g/ml was prepared. The solution was kept in a vial. One microliter of the sample was injected into the sim-dist GC using an autosampler.

#### 3.2.2.2 *Pour Point Testing*

The pour point of crude oil was tested following ASTM D97 by using Stanhope-Seta pour-point apparatus.

#### 3.2.2.3 *Density Testing*

Hydrometer was used to measure density of crude oil following ASTM D1298.

#### 3.2.2.4 *Wax Appearance Temperature (WAT) and Wax Dissolution Temperature (WDT) Determination.*

WAT (during cooling) and WDT (during heating) were measured by using a Perkin Elmer differential scanning calorimeter model USA, which was calibrated by pure indium as standard reference. Temperature scanning of crude sample was varied from 80 to -30°C in heating step and from -30 to 80°C in cooling step by using 5°C/min temperature changing rate (Chen *et al.*, 2004).

### 3.2.3 Crude Oil Fractionation

For crude oil separation, a novel method (Nguyen *et al.*, 1999) was modified by Srisirivilaikul (2004). The modified method was developed to reduce separation time by replacing p-xylene soxhlet extraction of asphaltene with n-heptane. As a reference, the solution was then cooled to 0°C and left overnight. Then filtration technique was employed to remove the asphaltenes as precipitate in the solution. After that acetone was added to the heptane solution and cooled to -21°C. The precipitate (or wax) was filtered and dissolved in 30 ml n-pentane. The solution was cooled to -21°C and allowed to stand overnight. Finally two fractions were obtained, the macrocrystalline waxes in the solution and microcrystalline waxes as precipitate. Both types of waxes were analyzed for hydrocarbons composition by Sim-Dist GC.

### 3.2.4 Effect of Solvents, Single Polymers, and Combined Polymer Inhibitors

#### 3.2.4.1 *Inhibitor Preparation*

The polymer inhibitors were prepared by 2 different ways. The first way was a solid polymer. It was chopped to tiny pieces before injected into crude oil. The second type was polymer solution (single and combined inhibitor in a solvent, mostly toluene) to get the homogeneous solution. The criterion of polymer

solution preparation was that solvent was used in small amount in order to avoid the effect of solvent on pour point testing.

#### 3.2.4.2 Determination of Inhibitor Efficiency

Pour point of crude after adding additives was tested. Pour point was important parameter to evaluate efficiency of each additive. The experiments in previous work (Numura, 2005) were conducted at 80°C, called “Previous Methodology”

##### *Previous Methodology (Numura, 2005):*

1. Weighed a calculated amount of wax inhibitor in a test cell
2. Added 25 grams of crude oil
3. Heat the test cell (80-85°C) until fluid was homogeneous and held at this temperature around 10 min\*
4. Measured the pour point of crude oil

\* *ASTM D97: Sample in the test cell is heated to 9°C above expected pour point*

Since the actual operating temperature in real field was 60°C, the new methodology was thus conducted at 60°C.

##### *New Methodology:*

The procedure was repeated the previous work except for third and fourth steps.

3. Heated the test cell to 60°C\*\* and held at this temperature around 15 min with stirring and then held at 60°C for 15 min without stirring
4. Measured the pour point of crude oil

\*\* *The actual operation of Lankrabue crude was at 60°C.*

The variation of concentration of polymer and solvent is shown in Tables 3.1 -3.2, respectively.

**Table 3.1** Concentrations of each polymer additives (each concentration based on pure polymer concentration excluding the solvent)

Concentration (ppm, wt/wt)	100	200	400	600	800	1,000	2000*
Weight of Additives (mg)	2.5	5	10	15	20	25	50

*\*This concentration was tested in some experiment.*

**Table 3.2** Concentration of each solvent

Concentration (%, wt/wt)	1*	2.5	5	7.5	10
Weight of Solvent (mg)	2.5	5	10	15	20

*\*This concentration was tested in some experiment.*

### 3.2.5 Inhibiting Stability Testing

The inhibiting stability of selected additive in crude oil was tested by keeping it at room temperature. The flow ability of crude was visually observed every day.