CHAPTER III
EXPERIMENTAL

3.1 Materials and Equipments

3.1.1 Equipments

- Canon Fenske type viscometer
- Zetasizer Nano apparatus, Model ZEN 3600, Malvern Instruments
- Bomb calorimeter
- Glass vial 15 mL
- Pipette and Auto-Pipette

3.1.2 Chemicals

3.1.2.1 Surfactants

Two types of nonionic surfactants were used in this work including; methyl oleate and palm oil methyl ester (POME). Their properties and structure are shown in Table 3.1

- Methyl oleate (MO), methyl ester, has carbon 18 atoms and a double bond. It was chosen because of the lowest amount of surfactant in microemulsion biofuels formulation. MO with 70% purity (technical grade) was purchased from Aldrich Chemistry Company.

- Palm oil methyl ester (POME) or biodiesel derived from palm oil was chosen because it has ester group as same as MO. The POME as B100 was received from Werasuwan Co., Ltd.

3.1.2.2 Cosurfactant

1-octanol was used in the research because it can work well with nonionic surfactant. It was purchased from Acros Organic. The chemical properties and structure of surfactant and cosurfactant are shown in Table 3.1.

3.1.2.3 Polar Phase

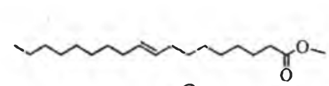
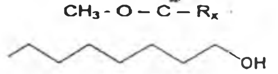
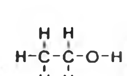
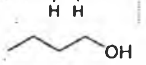
For this research, there are two types of alcohols which are ethanol and butanol. Their properties and structures are shown in Table 3.1.

- Ethanol with 95 % purity was used as viscosity reducer in microemulsion biofuels.
- Butanol with 99 % purity was used together with ethanol for improving the microemulsion biofuel property. Butanol was purchased from Aldrich Chemistry Company.

3.1.2.4 Oil Phase

Palm oil, RBDPO and diesel were used in this work. Food-grade palm oil and commercial-grade diesel were purchased from Morakot industries Public Company Limited and SHELL (Thailand) Public Company Limited, respectively. Moreover, refined bleached deodorized palm oil (RBDPO) was supported by Bangchak Biofuel Co., Ltd.

Table 3.1 Properties and structures of materials

Chemical	Stucture	Density (g/mL)	MW (g/mol)	Viscosity @40 °C (cSt)	Purity (%)
Surfactant and Cosurfactant					
Methyl Oleate (MO)		0.87	296.49	4.68	70
Palm oil methyl ester (POME)	$CH_3-O-C(=O)-R_x$	0.89	283.37	4.76	96.5
1-Octanol		0.83	130.23	6.06	99
Polar phase					
Ethanol		-	-	1.2	95
Butanol		-	-	3.64	99
Oil phase					
Palm oil	-	0.90	-	45.34	-
RBDPO	-	0.90	-	37.95	-
Diesel oil	-	0.82	-	4.1	-
B100	-	0.87		6.0	-

3.2 Experimental Procedures

The experimental procedure of this study is shown below

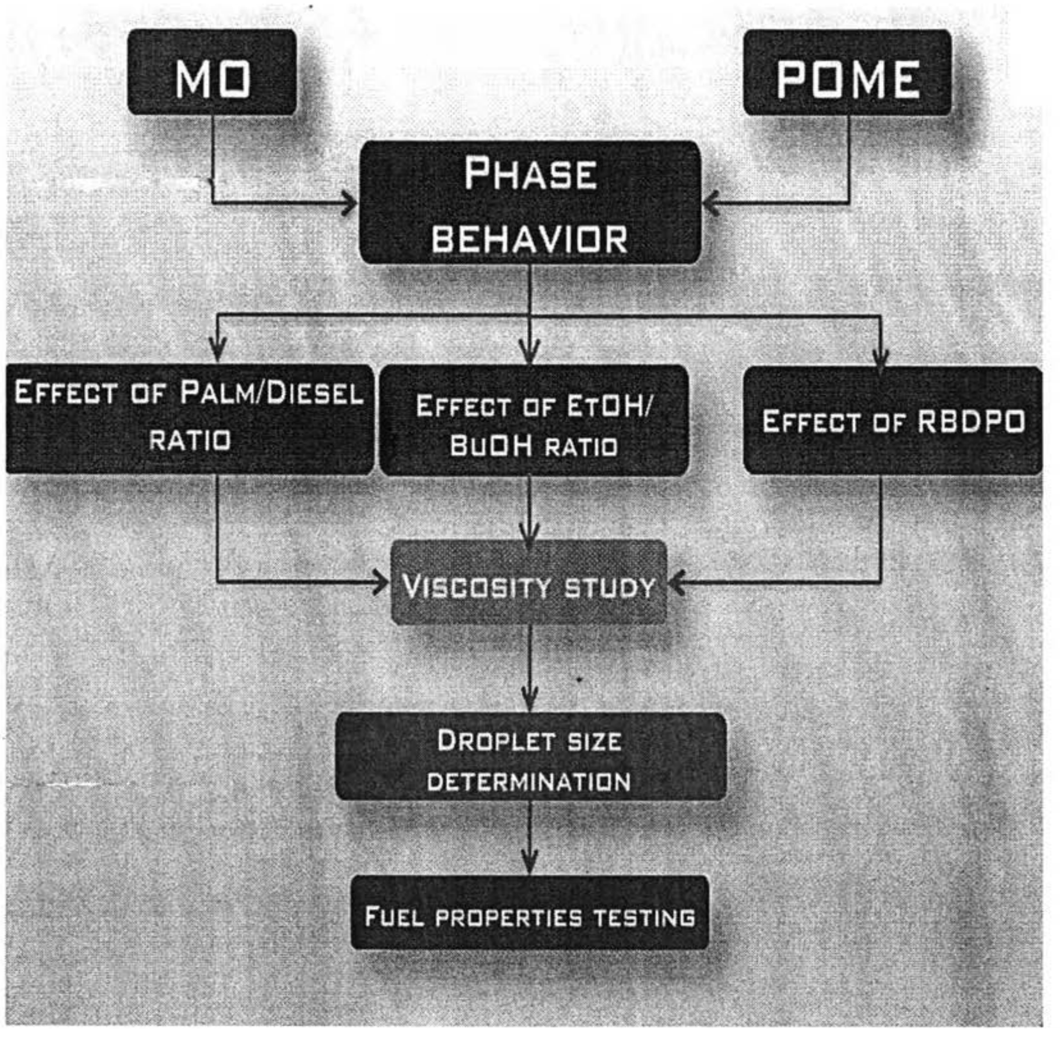


Figure 3.1 Overall procedure of this study.

3.2.1 Microemulsion Biofuels Preparation

1) The compositions of microemulsion biofuels consist of surfactant/cosurfactant blend at a molar ratio of 1:8, vegetable oil/diesel mixture was fixed at 1:1 by volume and the amount of polar phase (alcohol) was varied in each sample to studying the phase behavior. The samples were prepared in a 15 mL glass vial. All mixtures were hand-shaken and placed at the room temperature for 48 hr to reach equilibration. Phase behavior is determined by visual inspection with polarized light.

2) Different amounts of ethanol/butanol (vol.%, v/v) with palm oil/diesel blends (v/v) are added into surfactant-cosurfactant mixture to formulate reverse micelle microemulsions.

3.2.2 Phase Behavior Study

To study the phase behavior and miscibility of the microemulsion biofuels, a pseudo-ternary phase diagram is created. Pseudo-ternary phase diagram, consisting of six components in the system is investigated. In order to show the variations of the six-component mixture on the pseudo-ternary phase diagram, the top vertex of the triangle represents the surfactant/cosurfactant mixture at a constant ratio, while the two verticles at the bottom of the triangle represent the palm oil-diesel blends and the ethanol/butanol at left side and right side respectively. Lastly, total volume of the surfactant/cosurfactant mixture, the palm oil-diesel blends, and the ethanol/butanol is calculated to 100 % for all components. The miscibility curves are plotted as the boundary between the separate-phase and single-phase are. Area above the curve indicate isotropic phase systems, where a sufficient amount of the surfactant has been added to solubilize all of the components. The pseudo –ternary phase diagram is shown in Figure 3.2.

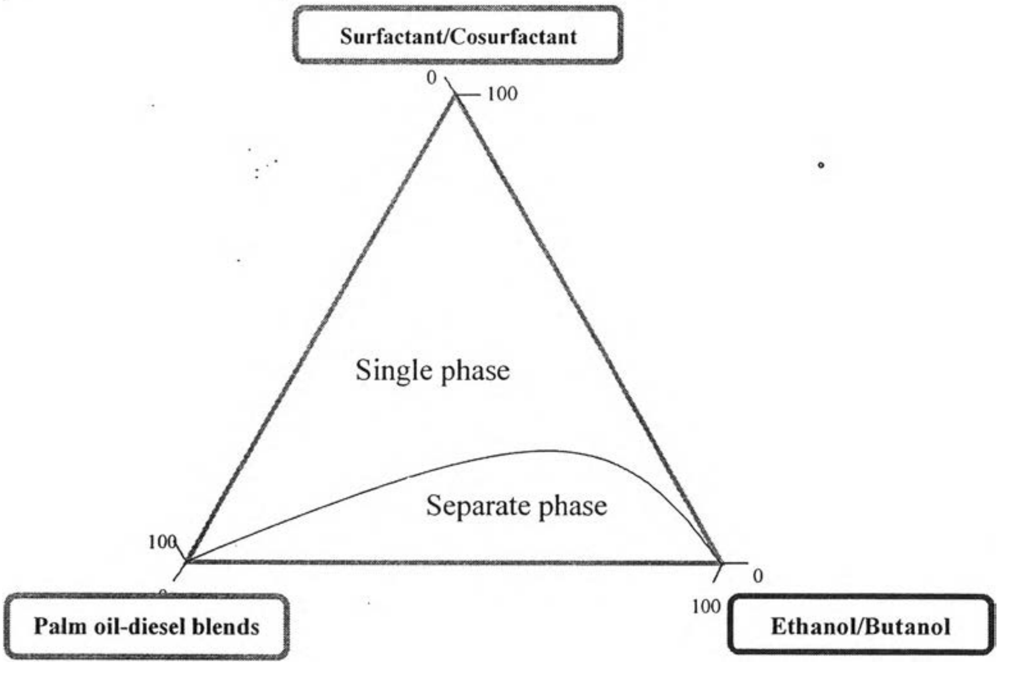


Figure 3.2 Pseudo-ternary phase diagram.

3.2.3 Kinematic Viscosity Measurement

1) The kinematic viscosity of microemulsion fuel was measured by Canon Fenske type viscometer followed ASTM D445.

2) A minimum sample volume of 7.0 mL of the microemulsion fuel (as recommended by the manufacturer) was then transferred into a viscometer chamber, and the time require for the fluid to flow between two specific points was measured. This study was measured at 40 °C.

3) The kinematic viscosity was calculated using Equation (3.1), which is provided by the manufacturer of the viscometer:

$$\nu = K_t T \tag{3.1}$$

where; ν is the kinematic viscosity (mm^2/s)

K_t is the viscosity constant at test temperature.

K_t can be calculated and is described in manufacture manual.

T is the efflux time (s) of the sample through the capillary tube.

3.2.4 Droplet Size Determination

1) Mean diameter (d_m) and size distribution of the microemulsion droplets were performed through Dynamic light scattering (DLS).

2) The measurements is performed at 25 °C at a fix angle of 173° (back scattering detection) by using a ZetasizerNano apparatus, Model ZEN 3600, Malvern Instruments.

3) The light source is a He-Ne laser (λ = 633 nm; 4mW) with a digital autocorrelation.

3.2.5 Fuel Properties of The Microemulsion Biofuel

The fuel properties of the microemulsion-based biofuel were determined to compare the properties between microemulsion biofuels and diesel No.2. The fuel properties were measured according to the American Standard Testing Methods (ASTMs). Table 3.2 shows the parameters and testing methods of the microemulsion-based biofuel. The parameters are the gross heat of combustion, cloud point, flash point, and kinematic viscosity. For this work, the fuel properties of the microemulsion-based biofuel were compared with those properties of No.2 diesel.

3.2.5.1 Gross Heat of Combustion

Gross heat of combustion was measured by an oxygen bomb calorimeter (model AC-350, LECO Corporation, USA) according to ASTM D 240. A crucible was used to place the fuels inside the calorimeter to test the heating value. The heat of combustion is calculated by the measured temperature increase of the water bath surrounding the bomb.

3.2.5.2 Cloud Point

In this study, the cloud point was determined following ASTM D 2500 by cooling bath, in which the test fuel was visually observed for the cloudiness and turbidity in cooling bath as the temperature was decreased every 1 °C.

Table 3.2 Parameters and testing methods of the microemulsion biofuel

Property	Method	Instrument
Gross heat of combustion (MJ/kg)	ASTM D 240	AC-350 Automatic Calorimeter
Cloud point (°C)	ASTM D 2500	Cooling bath
Kinematic viscosity at 40°C (mm^2/s)	ASTM D 445	Cannon fenske Kinematic Viscometer