CHAPTER III

METHODOLOGY

The experimental procedures for this study were divided into three parts. The first one involved the preparation of microemulsion biofuel from palm oil at different composition ratios and the generation of pseudo-ternary phase diagrams. The second part included an investigation of the properties of microemulsion biofuel, and the third part contained an examination of the performance of microemulsion biofuel in a real engine. A flow diagram of the experimental procedure is illustrated in Figure 3.1.

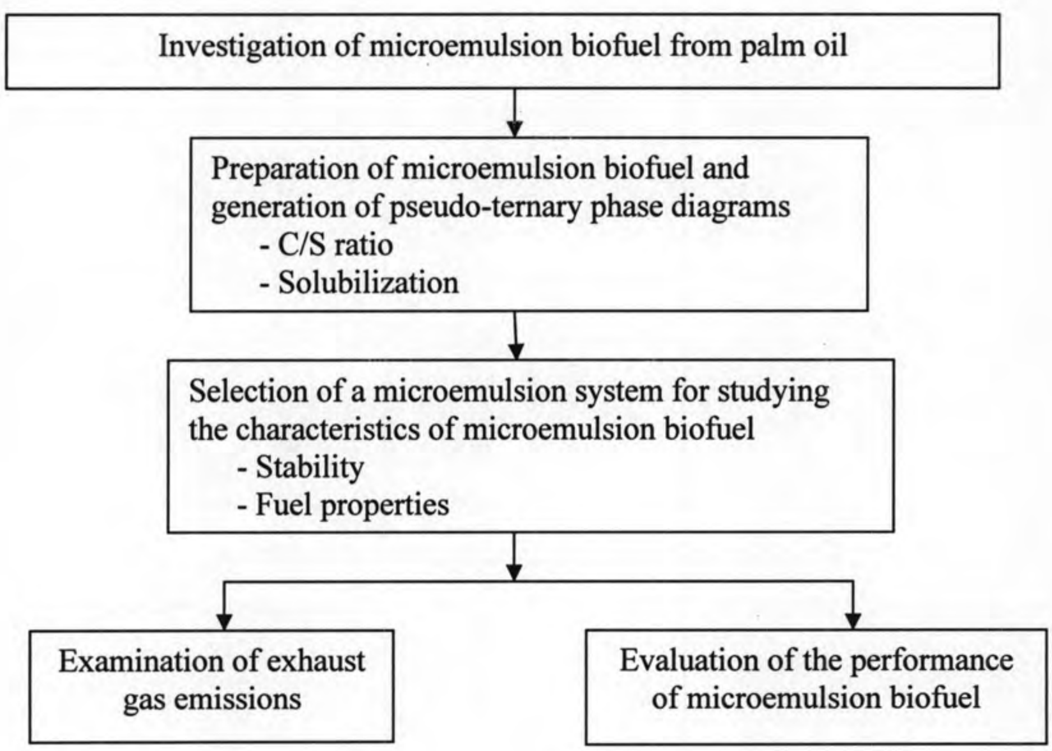


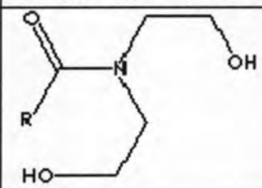
Figure 3.1 Flow Diagram of the Experimental Procedure

3.1 Chemicals and Materials

3.1.1 Surfactant

Coconut fatty acid diethanolamine (Comperlan KD) of 99.7% active strength was used as the nonionic surfactant in this study; it was purchased from Cognis Thailand Ltd. The properties of the surfactant are listed in Table 3.1.

Table 3.1 Physical and Chemical Properties of the Studied Surfactant

CAS No.	68603-42-9
Formula	$CH_3(CH_2)_nC(=O)N(CH_2CH_2OH)_2$
Chemical structure	
Molecular weight	280 - 290
Solubility in water	Soluble

Source: http://www.chemicalland21.com/specialtychem/perchem/COCAMIDE%20DIETHAN LAMINE.htm

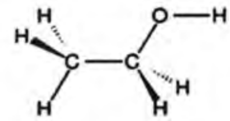
3.1.2 Palm Oil

Refined palm oil was used in this study. It was purchased from Lam Soon Co., Ltd. The fatty acid compositions of palm oil are shown in Table 2.1.

3.1.3 Cosurfactant

Absolute ethanol alcohol of 99.8% purity was used in this experiment; it was purchased from Italma Co.Ltd. The physical and chemical properties are listed in Table 3.2.

Table 3.2 The Physical and Chemical Properties of Absolute Ethanol Alcohol

Properties	Characteristics
CAS No.	64-17-5
Molecular Formula	C_2-H_6-O
Chemical Structure	
Appearance	colorless liquid
Melting point	-144 °C
Boiling point	78 °C
Flash point	14 °C
Water solubility	miscible in all proportions

Source: http://en.wikipedia.org/wiki/Ethanol

3.1.4 Water

Distilled water was used in this study for the preparation of microemulsion biofuel.

3.1.5 Petroleum Diesel

Petroleum diesel (i.e., conventional diesel) was used in this study; it was purchased from PTT Public Company Limited.

3.2 Phase Behavior Study and Pseudo-Ternary Diagrams

The phase behavior of the prepared microemulsion biofuel was studied in batch experiment. The microemulsion biofuel was prepared by mixing its four components: water, refined palm oil, the cosurfactant, and the surfactant, as shown in pseudo-ternary phase diagram (Figure. 3.2). A pseudo-ternary diagram describes the different phases existing in a system at different compositions. The cosurfactant and surfactant ratios (C/S ratios) in each pseudo-ternary diagram were kept constant, thus the four components could be represented with pseudo-ternary diagrams.

The experimental procedure for preparing microemulsion biofuel is as follows: weigh the cosurfactant and surfactant in a glass bottle at a 1:2 ratio (i.e., a C/S ratio of 0.5). Then mix it for 1 hour using a magnetic stirrer at 800 rpm to form a transparent solution. After that, weigh the refined palm oil and cosurfactant/surfactant in a 40 ml vial and mix them to form a homogeneous solution by using the magnetic stirrer for 1 hour at 800 rpm. Then add water in the solution and mix using the magnetic stirrer for 1 hour until the solution turns turbid and is no longer in the microemulsion phase.

The total weigh of the solutions (refined palm oil, cosurfactant/ surfactant and water) were 20 g and each component in the solution varied from 0% to 100% such as refined palm oil at 95%, cosurfactant/surfactant at 4.95%, and water 0.05%. This same procedure was performed with the other C/S ratios: 0.75, 1.0, and 1.25.

To determine the microemulsion phase area in the pseudo-ternary diagram the following components, C/S (absolute ethanol alcohol and coconut fatty acid dithanolamine), water (distilled water), and oil (refined palm oil), were varied. When a composition yielded a transparent homogeneous phase, it was considered capable of microemulsion. The component compositions that yielded microemulsion phases were plotted, as shown in Figure. 3.3. The shaded area represents the compositions in which microemulsions exist. Each composition in the pseudo-ternary diagram varied from 0% to 100%, by weight. The microemulsion phase obtained from this study was defined as microemulsion biofuel and called *MB100* in this study.

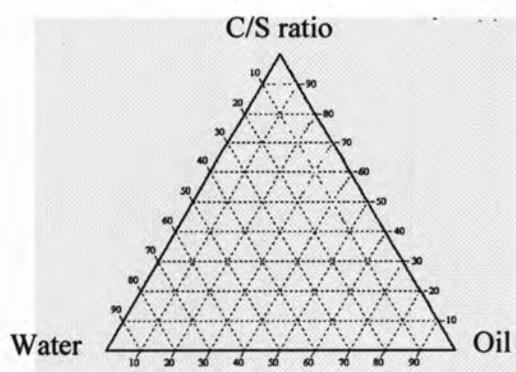


Figure 3.2 Pseudo-Ternary Diagram

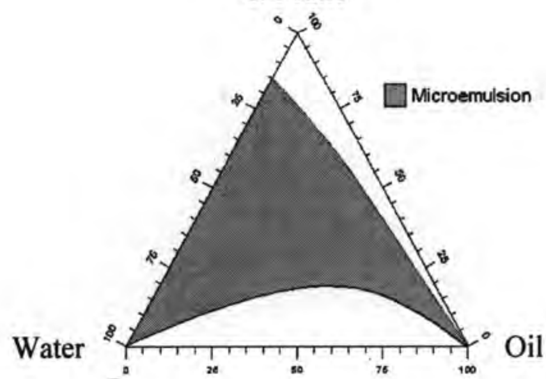


Figure 3.3 Determination of the Microemulsion Area

3.3 Stability Study of the Microemulsion Biofuel

Since nonionic surfactant was used for preparation the microemulsion biofuel, the stability within temperature range from 20 to 70 °C was tested for the selected compositions from the phase study. The compositions selected for this study were from microemulsion biofuel (MB100) prepared with the four C/S ratios: 0.5, 0.75, 1.0, and 1.25, three ratio of oil weight percentages of 95%, 90% and 85%, and for the water content from 0.05% and 0.1% for each oil composition. Moreover, the stability of microemulsion blending with diesel fuel at 20% and 5% microemulsion biofuel content by volume, called *MB20* and *MB5*, respectively. All of these compositions were kept in a water bath for seven days at 20, 35, 40, 45, 50, 55, 60, 65, and 70 °C to observe the physical stabilities.

3.4 Properties of the Microemulsion Biofuel and Its Blends

The homogeneous phase microemulsion biofuel obtained from 3.3 was selected for examination on the fuel's properties and its performance. The parameters tested for this study are acid number, carbon residue, cloud point, flash point, gross heat of combustion, kinematic viscosity, and copper strip corrosion. All of these properties were tested following ASTM (American Standard Testing Method) D664, D4530, D2500, D93, D240, D445, and D130, respectively. Gross heat of combustion and copper strip corrosion were parameters tested the performance of the fuel. The others parameters were tested to indicate if the fuel would affect the engine maintenance in the long term.

In addition to our microemulsion biofuel (MB100), its blending with petroleum diesel was also examined. The ratios of blending were 5% and 20% by volume of MB100 with petroleum diesel at 95% and 80%, called *MB5* and *MB20*, respectively. The microemulsion biofuels, MB20 and MB5, were prepared by mixing microemulsion (MB100) at 20% and 5% by volume with diesel fuel at 80% and 95% by volume, respectively in 1000 ml glass bottle, and stirring them for 1.5 hour until they formed homogeneous solutions. Then these solutions were kept by wrapping them with foil for protection against the oxidation reaction.

The testing of all parameters in this study was performed at the National Metal and Materials Technology Center (MTEC). The details of each parameter and its testing method are described in Table 3.3.

Table 3.3 The Parameters and Testing Methods of the Biodiesel

Parameters	**Instruments**	**Standard Testing Methods**[a]
Acid Number	Tirando Automatic Potentiometric Titratior	ASTM D664
Carbon Residue	ACR-M3 Micro Carbon Residue Tester	ASTM D4530
Cloud Point	ISL CPP 5Gs Cloud & Pour Point Tester	ASTM D2500
Flash Point	APM-7 Pensky-Martens Closed Cup Tester	ASTM D93
Gross Heat of Combustion	AC-350 Automatic Calorimeter	ASTM D240
Kinematic Viscosity @ 40 °C	Cannon mini-AV Automated Kinematic Viscometer	ASTM D445
Copper Strip Corrosion	SETA Model 11400-6 Copper/Silver Corrosion Bath	ASTM D130

[a]: American Standard Testing Method (ASTM international)

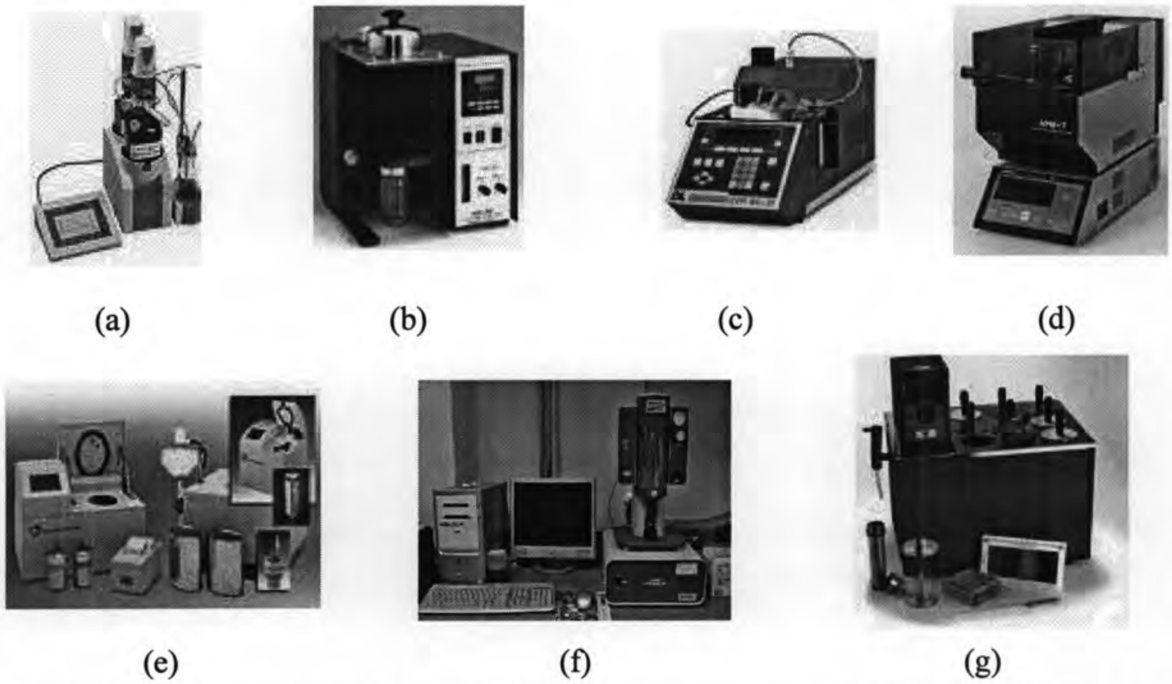


Figure 3.4 The Instruments for Analyzing Fuel Properties: (a) a Tirando Automatic Potentiometric Titratior; (b) an ACR-M3 Micro Carbon Residue Tester; (c) an ISL CPP 5Gs Cloud & Pour Point Tester; (d) an APM-7 Pensky-Martens Closed Cup Tester ; (e) an AC-350 Automatic Calorimeter; (f) a Cannon mini-AV Automated Kinematic Viscometer; and (g) a SETA Model 11400-6 Copper/Silver Corrosion Bath

3.5 Performance Study of Microemulsion Biofuel and Its Blends

The microemulsion biofuel, MB100, MB20, and MB5, were determined the performance of fuel compare with conventional diesel fuel. The fuels were run through single-cylinder engines.

3.5.1 Instruments for Testing

- Single-cylinder engine: KUBOTA, RT 100, 10 horsepower, as shown in Figure 3.5 and described in Appendix A.
- Performance Test Set: consisting of a 10 kW generator, electric lights, fuel consumption measurements, and temperature measurements (such as ambient temperature, water temperature, engine oil temperature, and exhaust temperature) as shown in Figure 3.6.

- Exhaust gas analyzer: HORIBA, MEXA-1600D as shown in Figure 3.7 and described in Appendix A.
- Smoke meter: TECNOTEST, 495/01 connected to a display screen (STARGAS, 898) as shown in Figure 3.8.
- Tachometer (cycle speed tester): LUTRON, DT-2234A as shown in Figure 3.9.

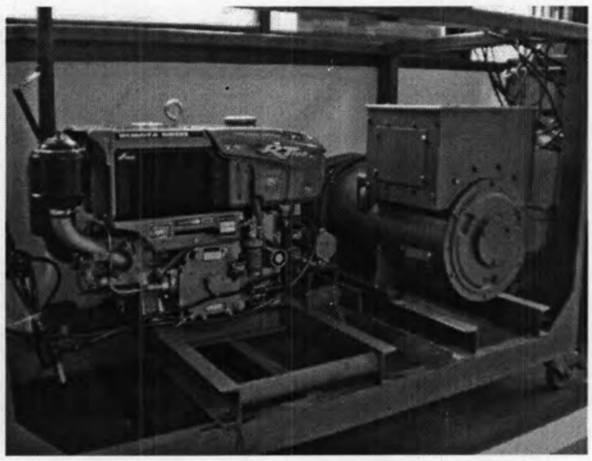


Figure 3.5 Diesel Engine Connect to Generator

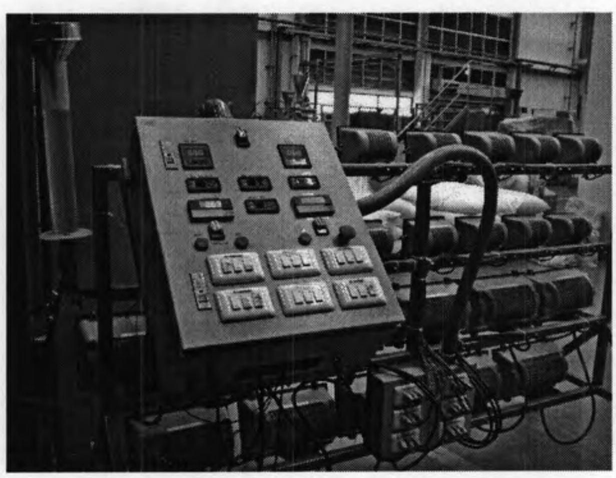


Figure 3.6 Electric Light, Fuel Consumption Measurement, and Temperature Measurement

Figure 3.7 Exhaust Gas Analyzer

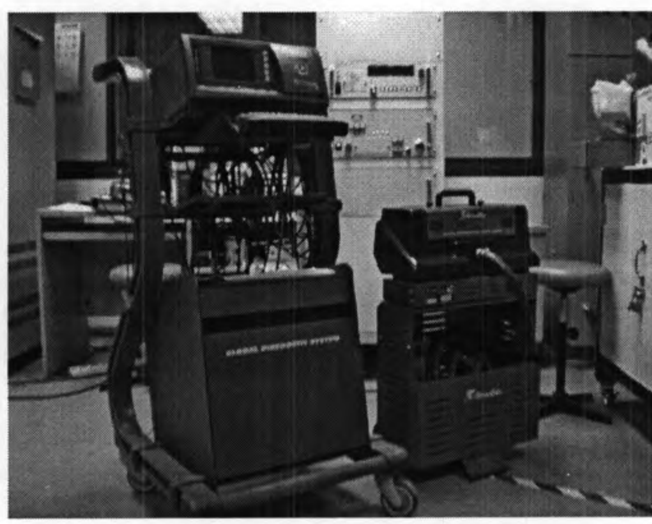


Figure 3.8 Smoke Meter

Figure 3.9 Tachometer

3.5.2 Testing Methods

The performance tests of microemulsion biofuel were tested with small motor engine, single-cylinder engine. Full load and full throttle testing were used to determine the power capacity (i.e., real engine performance) of the motor engine. At this time, nozzle was full-scale of the oil injection. Therefore the engine maximized oil and air consumption in order to carry load as large a load as possible.

The engine was connected to an electrical generator, which produced the current fed to electric lights. The total current which was utilized by a bunch of electric lights braked the engine's function. Hence, the lights that consumed more current (in terms of total watt increment) increased the load to the motor engine. The power engine could quantify from power supply by the principle of electromotive forces and the electric current.

Full load and full throttle testing were performed using maintaining the accelerator at the widest position; and took more load by opening light to decrease the initial testing cycled speed to around 2,400 rpm. Then, the more lights were turned on for a decrement of 200 rpm each step reaching 1,400 rpm. Each of parameter related to the performance of the engine, the production of black smoke, and the source of emissions was noted. The parameters and the testing methods used are described as follows:

- Power and Torque: testing from performance of engine which called power of electrical generator since generator have efficiency 80%. Therefore calculating of power and torque corresponds to Equations (1), (2) and (3):

$$P_{Gen.} = I \times V \quad (1)$$

$$P_{Engine} = \frac{I \times V}{\eta_{Gen}} \quad (2)$$

$$\tau = \frac{P_{Engine} \times 60}{2\pi N} \quad (3)$$

$$\lambda = \frac{AFR}{14.7} \quad (4)$$

Note: P_{Gen} is the electrical generator power (Watt)

I is the electric current (Ampere)

V is the electromotive force (Volt)

P_{Engine} is the engine power (Watt)

τ is the torque (Newton-meter)

N is the speed cycle (rpm)

η_{Gen} is the efficiency of the generator at the constant value 0.8

λ is the index of AFR

AFR or Air-fuel ratio is the mass ratio of air to fuel present during combustion. When all the fuel is combined with all the free oxygen, typically within a vehicle's combustion chamber, the mixture is chemically balanced and this AFR is called the stoichiometric mixture (often abbreviated to stoich). AFR is an important measure for anti-pollution and performance tuning reasons. Lambda (λ) is an alternative way to represent AFR (http://en.wikipedia.org/wiki/Air-fuel_ratio).

- Exhaust gas was measured using an exhaust gas analyzer. The exhaust gases are provided in Table 3.4
- Black smoke was analyzed using a smoke meter.
- Ambient temperature was the temperature in area testing engine
- Water temperature was the temperature in radiator of diesel
- Engine oil temperature was measured from the temperature of the lubricant in engine
- Exhaust temperature was temperature at exhausted nozzle
- Humidity was the humidity in the area of testing

Table 3.4 Exhausted Gas Analyses

Types of Exhausted Gases	**units**
Carbon monoxide (CO)	%
Carbon dioxide (CO_2)	%
Nitrogen oxide (NO_x)	ppm
Oxygen (O_2)	%
Total hydrocarbon (THC)	%
Ratio of air and fuel, AFR	-
λ (index of AFR when compare AFR at stoichiometry of diesel	-