CHAPTER 3
EXPERIMENTAL PROCEDURES

Biodiesel synthesized from palm oil and rice bran oil was investigated its kinetics under supercritical ethanol conditions. The study covered the effects of temperature, ratio of palm oil and ethanol. In this chapter, the details of chemicals, high pressure reactor system, and the analysis are discussed.

Two basic chemicals were used in the production of biodiesel. They were ethanol (99.8%) purchased from J.T. Baker Inc., USA, palm oil and rice bran oil grade vegetable cooking oil brand Morakot and King respectively.

The lists of chemicals below were used to verify our quantitative products.

3.1 Chemicals

1. Ethyl oleate 60% from Sigma-aldrich chemical company Inc., USA
2. Ethyl stearate 99% from Sigma-aldrich chemical company Inc., USA
3. Ethyl palmitate 99% from Sigma-aldrich chemical company Inc., USA
4. Ethyl linoleate 99% from Sigma-aldrich chemical company Inc., USA
5. Ethyl linolenate 98% from Sigma-aldrich chemical company Inc., USA
6. Ethanol 99.8% from J.T. Baker Inc., USA
7. Palm oil from Morakot Industry Co., Ltd., Bangkok "Morakot, มรกต"
8. Rice bran oil from Thai Edible Oil Co., Ltd., Bangkok "King, คิง"
9. Gas Hydrogen 99.99%, TIG, Thailand
10. Gas Helium 99.99%, TIG, Thailand

3.2 High pressure reactor system

A batch reactor containing 7 ml was constructed by stainless steel 316 and diameter 2.7 cm, thickness 1 cm, height 7.5 cm as shown in Figure 3.1. It was connected with pressure gauge ranging to 300 bar and monitored reaction temperature by thermocouple type K immerging directly into the reactor.

The reactor temperature was controlled by a furnace. A temperature controller ranging from room temperature to 1200°C was connected and heated the system. The system schematic is shown in Figure 3.2.

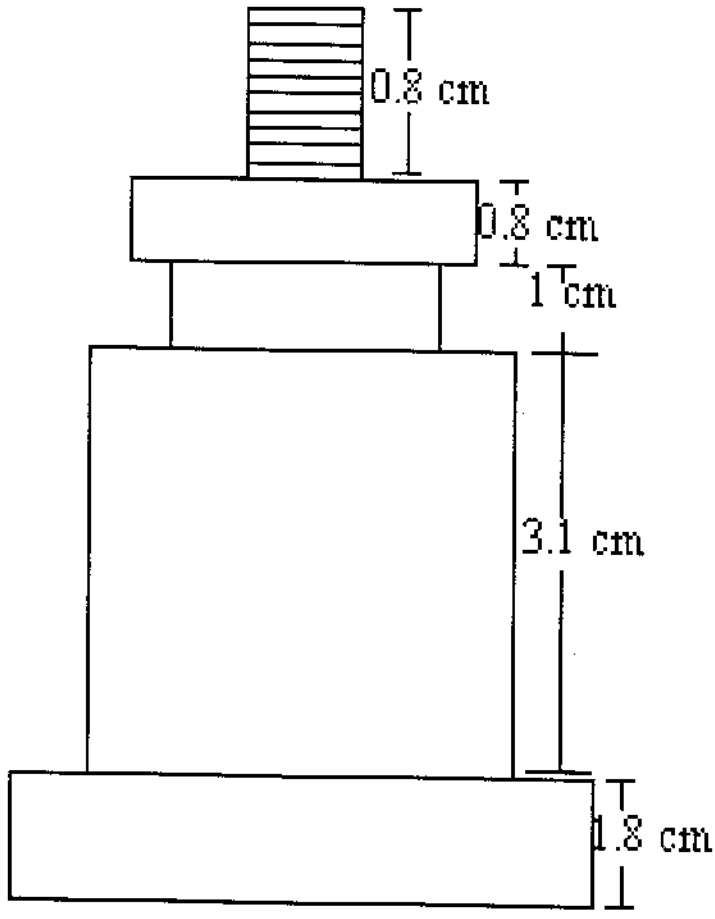

Figure 3.1 High pressure reactor.

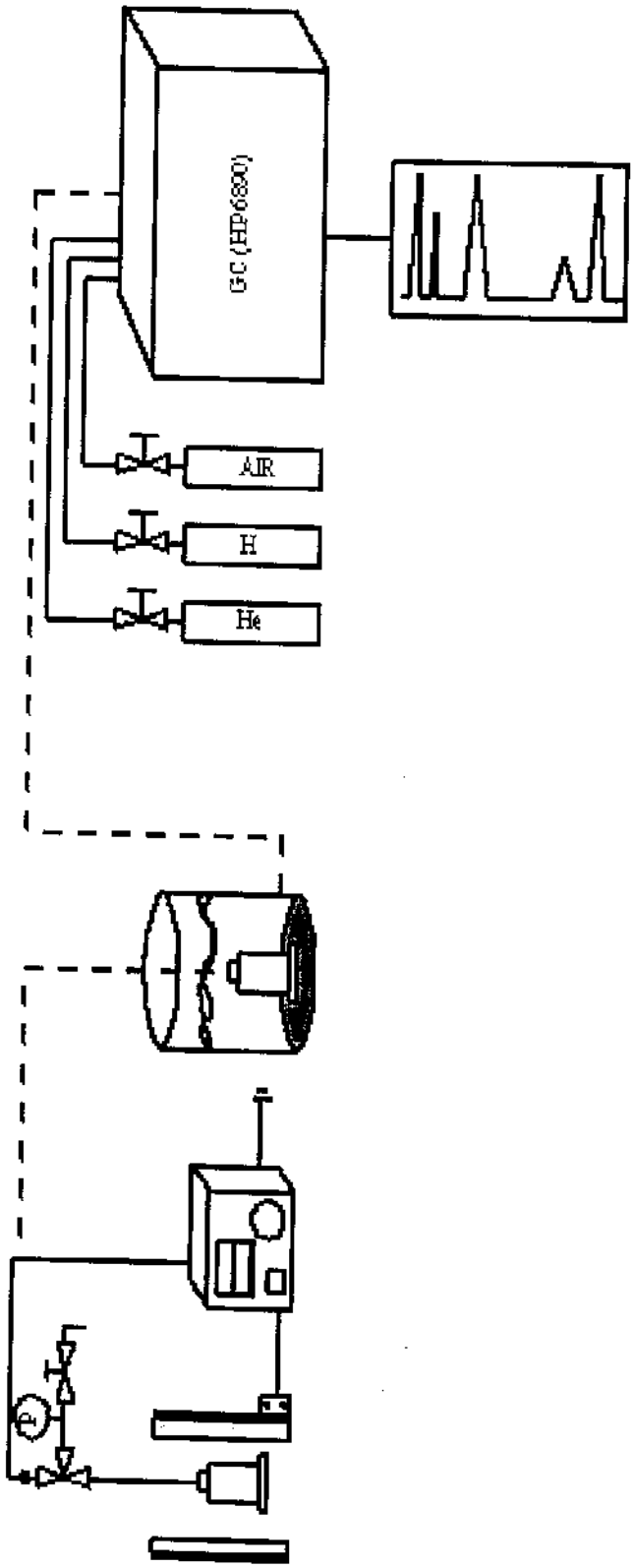

Figure 3.2 Supercritical ethanol biomass conversion system.

3.3 Reaction testing

The procedure of our study was presented as follows:

1. The batch type high pressure reactor was charged with a given amount of vegetable oil and liquid ethanol with a molar ratio of (oil:ethanol) 1:10-1:50. Since the reactor was small, the methodology to charge those material into a reactor was carried out by a syringe (10 ml).
2. The reaction vessel was then immersed into the furnace and kept for a set time interval for supercritical ethanol in 30-240 seconds.
3. After that, the reaction vessel was moved into the water bath to stop the reaction.
4. The treated vegetable oil was then allowed to settle for about 30 minutes to have the phase separated.
5. After that, the upper and the lower portion were analyzed for its composition by using gas chromatography (GC, HP 6890).

3.4 Biodiesel analysis

The upper and the lower portions were analyzed for its composition by using the gas chromatography (GC) (Hewlett-Packard, HP 6890) which consists of the column (HP-INNOWax (Cross-Linked PEG), 30 m x 0.53 mm x 1.0 μm, HP Part No.19095N-123) and flame ionizing detector (FID) operated at 45°C to 200 °C (5°C/minute) with 4 ml/minute flow rate of helium as a carrier gas. The sample injection volume was 1 μl and the peak identification was made by comparing the retention time between the sample and the standard compound.