

CHAPTER IV EXPERIMENT

4.1 Raw Material and Chemical

4.1.1 Polybutene-1 that used in experiments is the commercial grade for pipe was supplied from Basell Australia Inc. It was virgin plastic of 3-4 mm diameter pellets.

4.1.2 The catalyst used in this study was an iron supported on activated carbon. It was grounded to around 0.3 mm

4.1.3 Hydrogen gas (purity 99.5% minimum)

4.1.4 Toluene (commercial grade; purity 80% minimum)

4.2 Apparatus and Instrument

4.2.1 The reaction of polybutene-1 with Fe/activated carbon was carried out by using apparatus (shown in Figure 4.1). The microreactor is a stainless steel tube SS.316 with an inner volume of 70 cm³, 30 mm inside diameter, (shown in Figure. 4.2). It was heated by an electricity 450 watt, and the temperature was measured by thermocouple type R size of 1.6 mm diameter. The temperature was controlled to an accuracy of ± 5 °C by means of a programmable temperature controller. The shaking of microreactor was controlled by a speed motor. The separation of liquid oil from solid by the vacuum filter (shown in Figure 4.3). The boiling ranges of oil yield were determined by used Gas Chromatograph (GC Simulated Distillation) with the following conditions : Open Tubular Columns type capillary columns : RTX2887 , initial temperature 40 °C, final temperature 350 °C , FID detector , sample size 0.5 μ L, carrier gas helium , programming rate 10 °C/min.

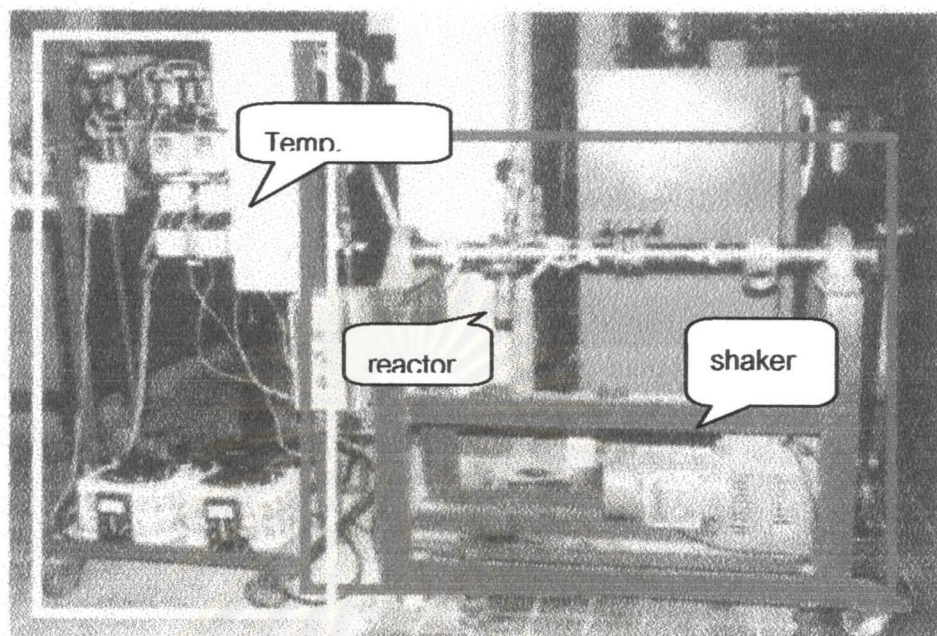


Figure 4.1 The reaction experimental unit for conversion of Polybutene-1 into oil products using Fe/AC catalyst.

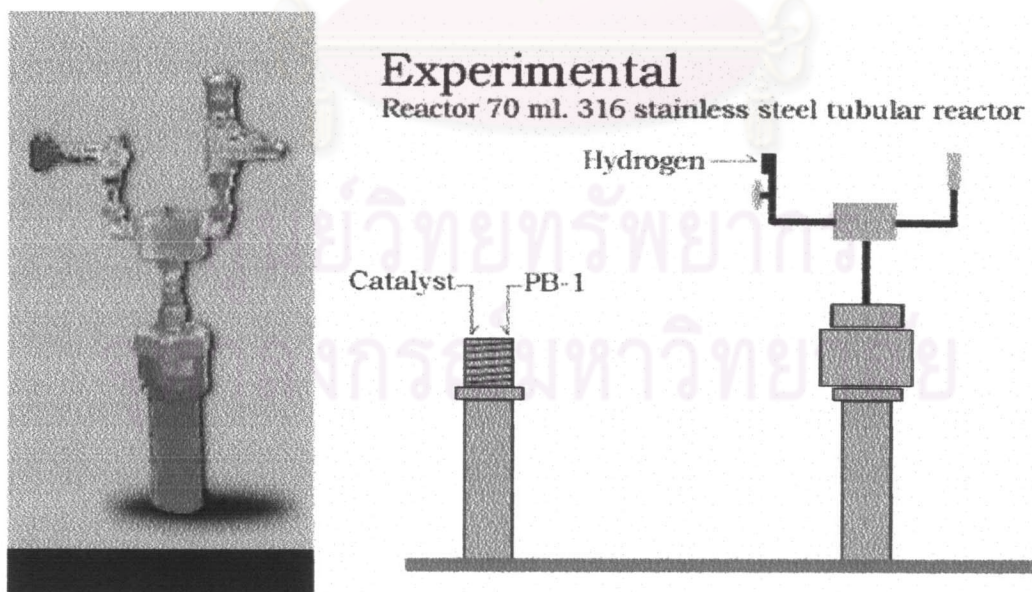


Figure 4.2 The microreactor

4.2.2 Vacuum pump

The separation of liquid oil from catalyst and residue by used the vacuum filter (shown in Figure 4.3).

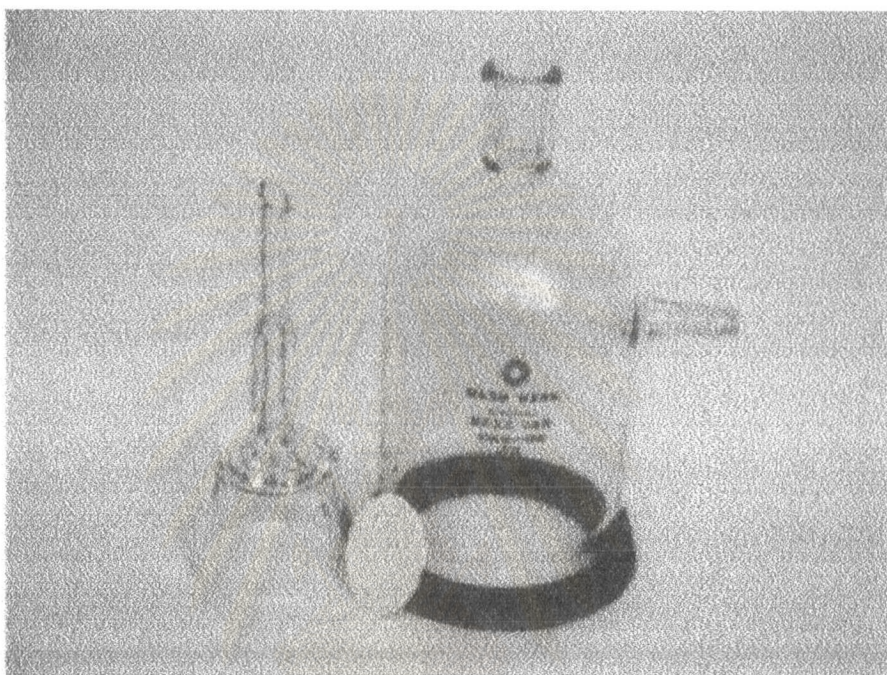


Figure 4.3 The vacuum filter

4.2.3 Gas Chromatography (GC Simulated Distillation)

The oil composition analysis used Gas Chromatography, Perkin Elmer as ASTM D 2887, analyzing the boiling range distribution is simulated by the use of gas chromatograph (GC Simulated Distillation) at PTT Research and Technology Institute followed ASTM D2887-93 method (Modified Method by PTT Research and Technology Institute). A nonpolar packed or open tubular (capillary) gas chromatographic column is used to elute the hydrocarbon components of the sample in order of increasing boiling point. The column temperature is raised at a reproducible linear rate and the area under the chromatogram is recorded throughout the analysis. Boiling points are assigned to the time axis from a calibration curve obtained under the same chromatographic

conditions by analyzing a known mixture of hydrocarbons covering the boiling range expected in the sample. The boiling range distribution can be obtained.

4.3 Processes of the Experiment

4.3.1 The processes prepared for catalyst shown as follows :^[9]

A preparation of iron on activated carbon has been done through impregnation method with different percentage of iron at 1, 5 and 10% about 200 g of each type of catalyst are prepared with the following steps :

4.3.1.1 Injecting distillate water at an equal level of a height of activated carbon in a container before allowing it to enter in vacuum equipment for 3 hours and finally air bubble will be found on its surface.

4.3.1.2 Impregnation with aqueous solution of ferric nitrate ($\text{Fe}(\text{NO}_3)_3$) with required percentage of iron on deaerated activated carbon at room temperature for 2 hours.

4.3.1.3 Drying slurry with vacuum rotary evaporator to a temperature of 80°C for 3 hours, and then dry and powdered activated carbon with $\text{Fe}(\text{NO}_3)_3$ which is in pore putting it in an for baking overnight to a temperature of 120°C .

4.3.1.4 The next day, calcination activated carbon with $\text{Fe}(\text{NO}_3)_3$ which is in pore to temperature of 450°C for 3 hours for driving off nitrate, iron on surface of activated carbon will be yielded.

4.3.1.5 Reducing catalyst with hydrogen to a temperature of 400°C for 1 hour and refilling presulfation with hydrogen sulfide to a temperature of 450°C for 1 hour and keeping it cool at room temperature, catalyst usable for intended purposes will be given.

4.3.2 Procedure of the experiment

Procedures of the experiment are shown in figure 4.4.

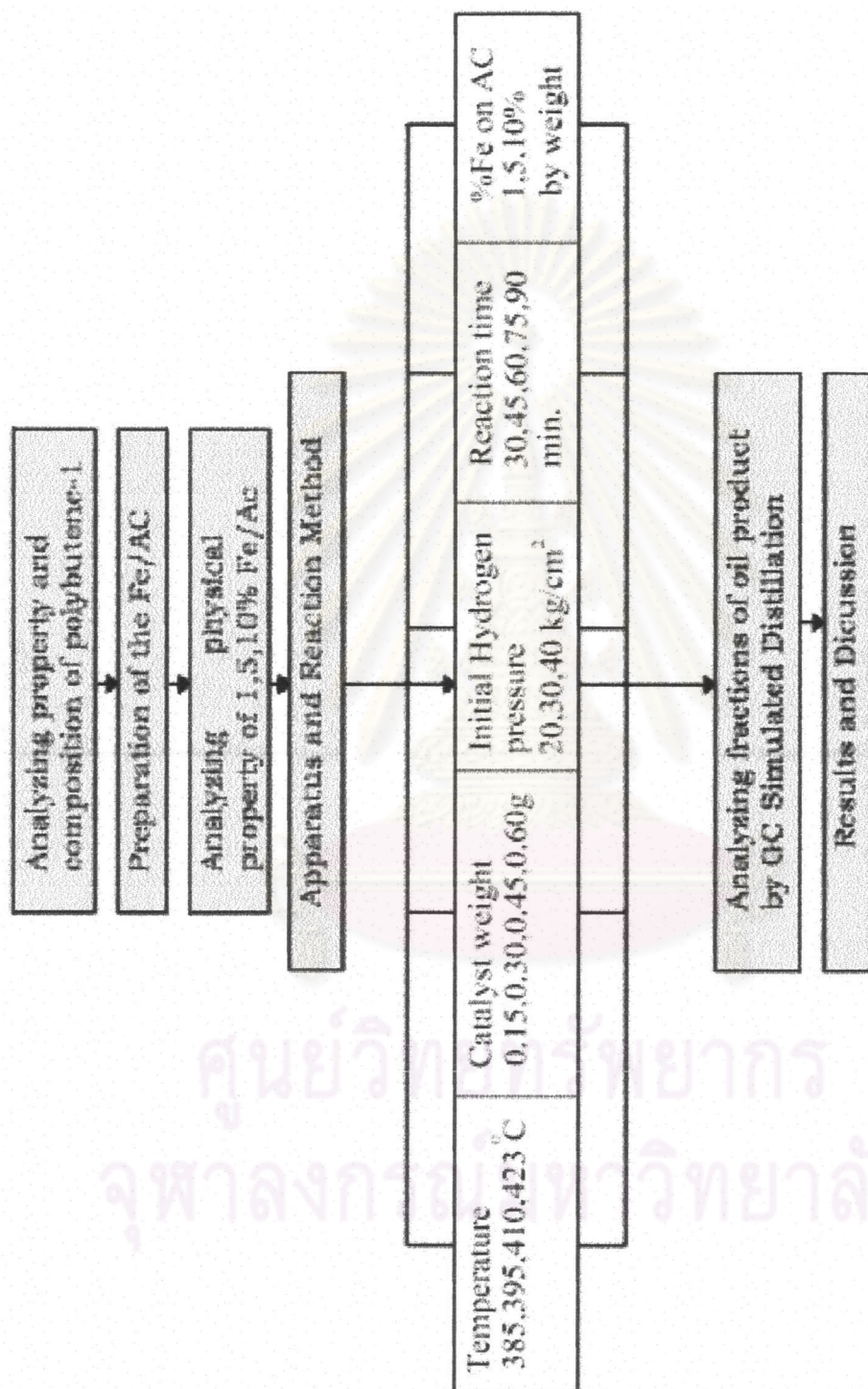


Figure 4.4 Experiment scheme of the hydrocracking of polybutene-1 by Fe/AC catalyst

4.3.2.1 Weighing 15 g of polybutene-1 and 0.3, 0.45, and 0.60 g of catalyst and putting it in 70 ml-reactor.

4.3.2.2 Compressing hydrogen gas at its pressure of 20, 30, 40 and 50 kg/cm² and checking its leakage.

4.3.2.3 Putting reactor on heater coil, covering insulator, fixing thermocouple for the measurement of internal and external temperatures of reactor and finally setting its reaction temperature of 385, 395 410 and 425 °C.

4.3.2.4 Fixing reactor with shaker at 120 rpm when its reaction corresponds to a time set from 30, 45, 60 75 and 90 min.

4.3.2.5 Cooling a reactor by removing insulator and heater, blowing it with an electric fan and keeping it cool until temperature goes down to a room temperature.

4.3.2.6 Filtering yielded oil under vacuum condition, keeping some of it for analysis by distillation gas chromatography to find out oil composition and filtering all of yield products by using toluene as a solvent of products remaining in the reactor. Remainders in the filter are catalyst and coke or solid without any changes into the products. As a result, the data obtained from this processes are usable for calculating conversion percentage.

4.3.3 The product composition analysis by Gas Chromatograph

The boiling ranges of oil yield were determined by Gas Chromatograph (GC Simulated Distillation) at PTT Research and Technology Institute for naphtha (IBN-200 °C), kerosene (200-250 °C), gas oil (250-300 °C), and long residue (up to 350 °C).

4.3.4 The catalyst surface analysis after using in reaction

4.3.4.1 Surface morphology using Scanning Electron Microscopy

4.3.4.2 Surface area using BET