CHAPTER III
EXPERIMENTAL

3.1 Material

3.1.1 Chemicals

- $(NH_4)_6Mo_7O_{24}\cdot4H_2O$ was purchased from Carlo Erba.
- $Ni(NO_3)_2\cdot6H_2O$ was obtained from Carlo Erba.
- $Pt(NH_3)_4(NO_3)_2$ was purchased from Sigma-Aldrich.
- γ–Alumina support was produced by Saint–Gobain Norpro Corporation.
- HiSil 255 SiO_2 support was obtained from PPG Company.
- KF–840 $NiMo/Al_2O_3$ catalyst (2.5 wt% Ni, 12.7 wt% Mo, and 6.4wt% P_2O_5) was obtained from Akzo–Noble.
- Ethylbenzene (98% purity) was obtained from Carlo Erba.
- *p*–Methylethylbenzene was purchased from Fluka.

3.1.2 Gases

- N_2 (99.99 % Purity)
- H_2 (99.99 % Purity)
- He (99.99 % Purity
- Air Zero (99.99% Purity)

3.2 Equipment

The equipments used in this work are listed as follows.

- A Continuous flow fixed bed reactor consisting of a high pressure pump, mass flow controllers, back pressure regulator, and PID temperature controller equipped with a thermocouple (Type K).
- Agilent Technologies model 6890N gas chromatograph with flame ionization detector equipped with HP–5 column.
- pH-meter

3.3 Methodology

3.3.1 Catalyst Preparation

3.3.1.1 The Pt/SiO_2 catalyst containing 1% wt.% Pt was prepared by means of impregnation method of SiO_2 support with aqueous solution of $Pt(NH_3)_4(NO_3)_2$. After drying at 110°C overnight, the catalyst was calcined in flowing air at 400°C for 2 hours.

3.3.1.2 The $Mo/\gamma-Al_2O_3$ catalyst containing 12.7 wt.% of Mo was obtained by impregnation of $\gamma-Al_2O_3$ with aqueous solution of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$. Firstly, the $\gamma-Al_2O_3$ was dried at 110°C for 12 hours. After that the $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ precursor solution was slowly dropped on $\gamma-Al_2O_3$ support. Next the sample was left at room temperature for 2 hours before drying at 110°C overnight. Lastly, they were calcined in air at 500°C for 6 hours.

3.3.1.3 The 2.5 wt.% $Ni/\gamma-Al_2O_3$ catalyst was prepared by means of impregnation technique of the $\gamma-Al_2O_3$ carrier with aqueous solution of $Ni(NO_3)_2 \cdot 6H_2O$. After drying at 110°C overnight, the catalyst was calcined in flowing air at 400°C for 2 hours.

3.3.1.4 The $NiMo/\gamma-Al_2O_3$ catalyst containing 12.7 wt.% Mo and 2.5 wt.% Ni was obtained by co-impregnation of $\gamma-Al_2O_3$ with aqueous solution of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ and $Ni(NO_3)_2 \cdot 6H_2O$. After drying at 110°C overnight, the catalyst was calcined in flowing air at 500°C for 4 hours.

3.3.2 Catalyst Characterization

The pH of the catalyst/water slurry was measured by using a pH electrode (Denver Instrument model UP–5). A total of 50 mg of catalyst dispersed in 15 ml of deionized water was used in the measurement, and the measurement continued until a constant pH level was achieved (Arvela *et al.*, 2007)

3.3.3 Catalytic Activity Testing

Because of *para*–methylethylbenzene (AR–grade) is expensive, in order to reduce the cost of the research, ethylbenzene which has one ethyl group connected to the aromatic ring, similar to the structure of methylethylbenzene was used as a preliminary model feed for hydrogenolysis reaction to get an optimum condition before applying it to the hydrogenolysis of *p*–methylethylbenzene (*p*–MEB).
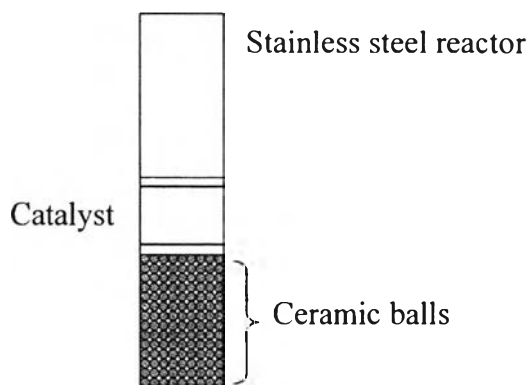
Figure 3.1 Schematic of the reactor and catalyst loading.

The hydrogenolysis reaction was conducted in a continuous flow fixed bed reactor, with 15 mm outside diameter and 480 mm length. The catalyst was packed at the center of the reactor over ceramic balls, which were used as catalyst bed supporter. Before carrying out the catalytic measurements, the catalyst was reduced in flowing hydrogen. The schematic of the reactor was presented in Figure 3.1. The temperature of catalyst bed was monitored and controlled by a PID temperature controller equipped with a thermocouple (Type K). Liquid reactant was delivered to the catalytic device by means of high-pressure pump (Gilson 307). The hydrogen flow and the total pressure were controlled by a mass flow controller (Brooks 5850 TR) and a back pressure regulator (Siemens model 63SU-L), respectively. The liquid product was trapped in a condenser while gas product was trapped in gas sampling bag. The products were collected hourly. The schematic of the reaction system is shown in Figure 3.2. The collected liquid product was qualitatively and quantitatively analyzed by a 6890 Agilent gas chromatograph equipped with a flame ionization detector (FID) and HP–5 column.
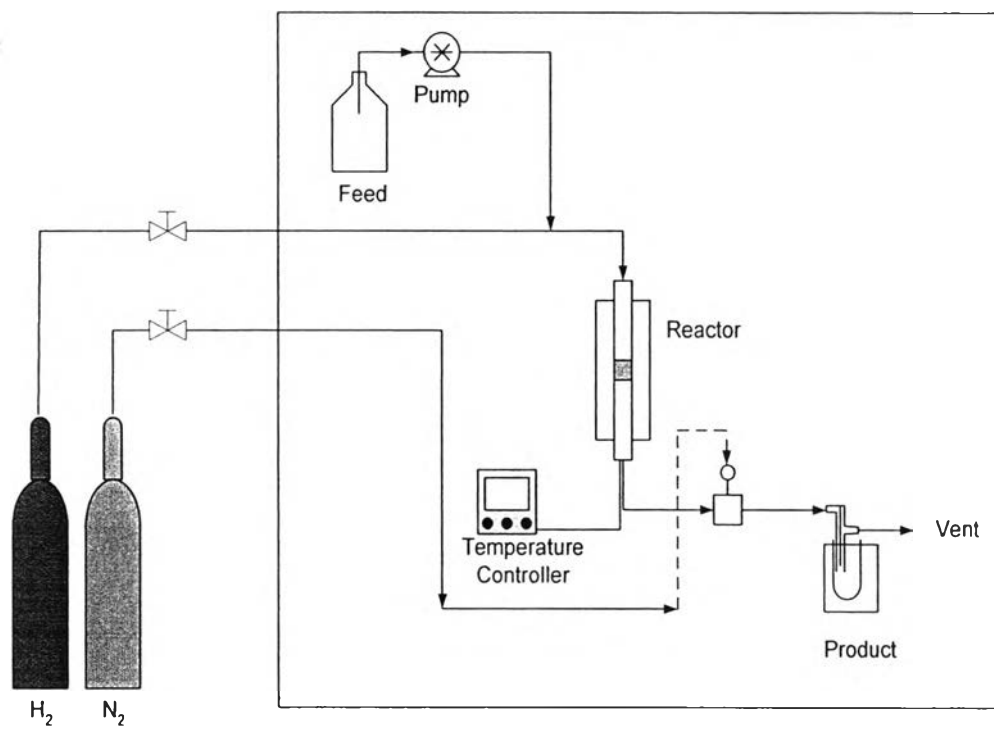
Figure 3.2 Schematic of the reaction system.