CHAPTER III EXPERIMENTAL

3.1 Materials

- 3.1.1 The model plastic of Low Density Polyethylene (LDPE) used in this study as commercial grade (code: LL 0180) was obtained from TPI Co., Ltd.
- 3.1.2 The coal used was lignite from Mae Moh Lumphang province as a powder of ~ 60 mesh. An analysis of lignite (ultimate and proximate method) is given in Table 4.1.
- 3.1.3 Hydrogen gas (purity 99.5% minimum): industrial gas from T.I.G. Trading Ltd.
- 3.1.4 Toluene (commercial grade; purity 80% minimum)
- 4.1.4 The catalyst used in this study was a commercial grade NiMo/Al₂O₃, composed of 2.72 wt% Ni and 13.16% Mo. The catalyst, ground to ~60 mesh, was presulfided external to the reactor prior to use. The procedure for presulfiding NiMo/Al ₂O₃ began by predrying NiMo/Al₂O₃ in flow of N₂ for 1 h. at 300°C then a 10 vol% H₂S/H₂ gas mixture was flowed over the catalyst at 225°C for 1 h followed by 315°C for 1 h and 370°C over NiMo/Al₂O₃ for 1 h after which the catalyst was then allowed to cool to room temperature under a N₂ flow.[16]

3.2 Apparatus and Instrument

3.2.1 The reaction of low-density polyethylene with coal was converted to fuel oil by using apparatus shown in Figure 4.1. Apparatus for reaction experimental unit shown in Figure 4.1

- PID (Proportional Integral Differential Controller)
- VARIAC
- Microreactor (70 ml of volume)
- Motor



Figure 3.1: The reaction experimental unit for catalytic coprocessing of low density polyethylene with lignite using Ni-Mo on alumina

3.2.2 Gas Chromatography (GC Simulated Distillation)

Perkin Elmer as ASTM D 2887; Analyzing the boiling range distribution determination by distillation is simulated by the use of gas chromatograph (GC Simulated Distillation) at PTT Research and Technology Institute followed as ASTM D2287-93 (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 cuve 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.

- 3.2.3 Vacuum pump (separation oil product from catalyst and residue)
- 3.2.4 Fourier- Transform IR spectrometer:

model 1760X, Perkin Elmer; analyzing functional groups of oil products at Scientific and Technological Research Equipment Center Chulalongkorn University (STREC)

3.2.5 CHNS/O Analyser:

Model PE2400 series II, Perkin Elmer, Analyzing by Pyrolysis in High-Purity Oxygen (Static-State Oxidation) Gas Chromatographically Separated by Frontal Analysis Quantitative Detected by Thermal Conductivity Detector.

3.3 Reactions and procedures

Reactions were performed for systems of LDPE/lignite to evaluate the product distribution, the effect of reaction temperature, reaction pressure, reaction time, and ratio of LDPE to lignite coal on the reactivity. Each experiment was used 15 g of low-density polyethylene and 0.45 g presulfided NiMo/Al₂O₃ powder pour in reactor, the reactions were conducted in 70 cm³ stainless steel tubular microreactor. The conditions are given in Figure 3.2 by fixing constant shaking the reactor with frequency of 108 Hz. After reaction, the reactor was quenched in water. Each experiment was used 15 g of LDPE, 0.5-5.0 g of coal. Total loading of sample was not over than 20 g.

The low-density polyethylene with coal was converted to oil product under the following conditions:

- Reaction temperature at 400, 420, 435, 450 and 480°C
- Pressure of hydrogen at 30, 45, 60 and 75 kg/cm²
- Reaction time 30, 45, 60, 90, 120 and 180 minutes
- Ratio of LDPE with lignite coal at 15:0.5, 15:1, 15:2.5, 15:3 and 15:5



Figure 3.2: Experiment scheme of the production of catalytic coprocessing of low density polyethylene with lignite coal.

After the reaction, products gases were determined by weighing the tubular microreactor before and after the release of gaseous products; this fraction in the product distribution is called gas. The liquid products were brought to filtration step. The used coal and catalyst were separated from liquid products by vacuum pump. The liquid products were analyzed by Simulated Distillation Analysis. The boiling point range of the liquid from the different reactions was obtained by Simulated Distillation follow as the ASTM D-2887 method. Additional replications were performed on a random basis. After the entire set of reactions was completed, the reactions were replicated, since these reactions produced marked changes in the yield of the reactants and in the amount of gas.

After the Simulated Distillation recovery was determined, the weight percentages of liquid products boiling in the ranges ibp-100 (where ibp is the initial boiling point), < 200, <300, <400, and 500 °C were calculated. Standard deviations for these weight percentages were calculated using standard deviations obtained in the product distributions and from Simulated Distillation recovery. The total product distribution of the reaction product was calculated by the Simulated Distribution.