CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Used Lubricating oil was obtained from Gasoline Station II Directorate of Welfare, Royal Thai Air Force.

3.1.2 Hydrogen gas (purity 99.5% minimum)

3.1.3 Toluene (commercial grade; purity 80% minimum) from Lab Systems Co.,Ltd.

3.1.4 The catalyst used in this study was a commercial grade Ni-Mo/Al_2O_3 and HZSM-5 catalyst.

- The Ni- Mo/Al_2O_3 catalyst was grinded in to 600°C for 4 hours before used.
- HZSM-5 catalyst is ready to use.

3.2 Apparatus and Instruments

3.2.1 The used lubricating oil was converted to light oil by using apparatus shown in Figure 3.1.

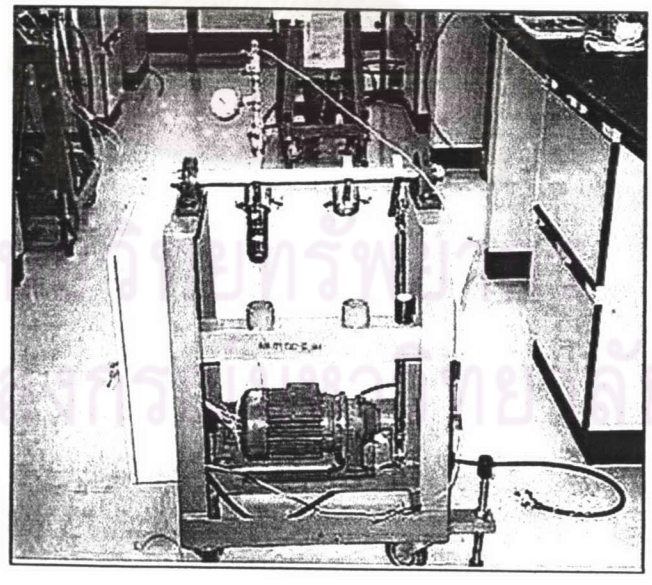

Figure 3.1 Apparatus for reaction experimental

- Microreactor (70 ml of volume) shows in Figure 3.2

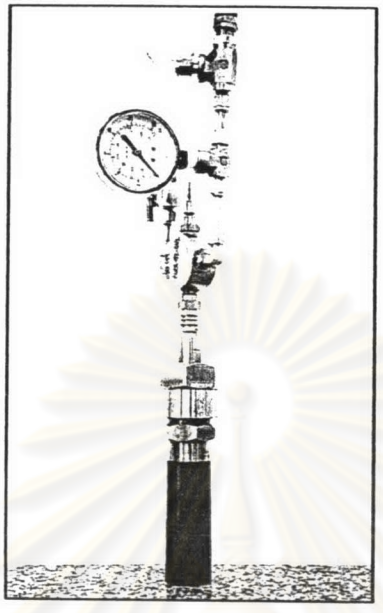

Figure 3.2 Microreactor for reaction experimental

3.2.2 Simulated Distillation Gas Chromatography (DGC) shows in Figure 3.3

Perkin Elmer as ASTM D 2887; Analyzing the boiling distribution determination by distillation is simulated by the use of Simulated Distillation Gas Chromatography at Fuels Research Center Chemical Technology Chulalongkorn University . Anopolar 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 temperture is raised at a reproducible linear rate and area under the chromatogram is recorded thrughout the analysis. Boiling points are assigned to the time axis from a calibration cure abtained under the same chromatographic conditions by analyzing a know mixture of hydrocarbons convering the boiling range expected in the sample. The boiling range distribution can be obtained. The apparatus shows in Figure 3.3.

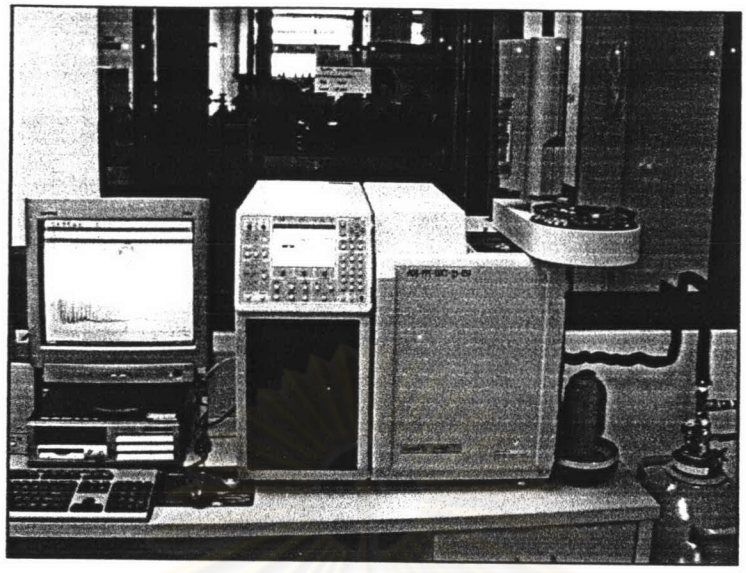

Figure 3.3 Simulated Distillation Gas Chromatography (DGC)

3.2.3 Vacuum pump (separation oil product from catalyst and residue) shows in Figure 3.4

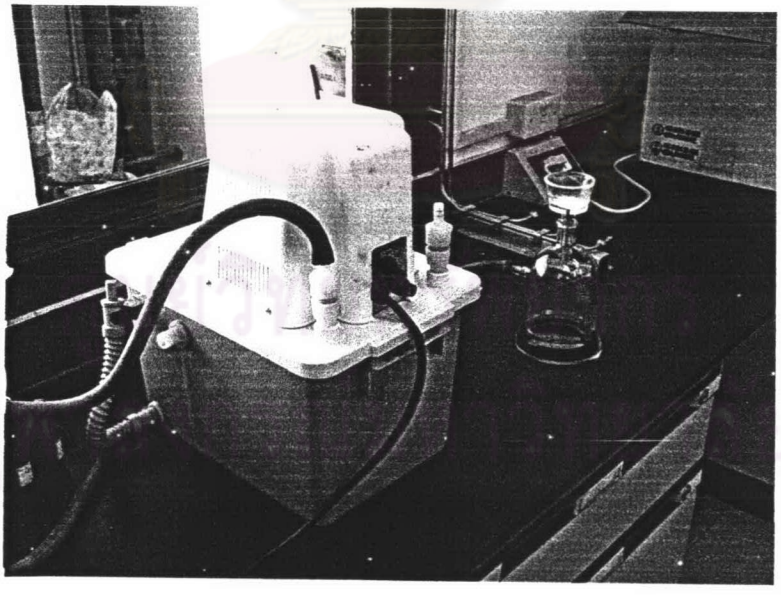

Figure 3.4 Vacuum pump

3.3 Reactions and procedures

The reactions were performed in a system of used lubricating oil to evaluate the product distribution. The effect of reaction temperature, reaction pressure, reaction times, and percentages of catalyst were investigated. Each experiment was used 20 g of used lubricating oil and catalysts to stipulate a condition.

The used lubricating oil was converted to light oil product under the following conditions:

- Reaction temperature range from 400 to 470^{0}C
- Pressure of hydrogen range from 0 to 200 psi
- Reaction time:
    - Range from 30 to 120 minutes in case of Ni-Mo/Al_2O_3.
    - Range from 30 to 90 minutes in case of HZSM-5.
- Quantity of catalyst as percent by weight:
    - Range from 0 to 5% in case of Ni-Mo/Al_2O_3.
    - Range from 0 to 0.6% in case of HZSM-5.

The experimental schemes of two catalysts were shown in figures 3.5 and 3.6 respectively.

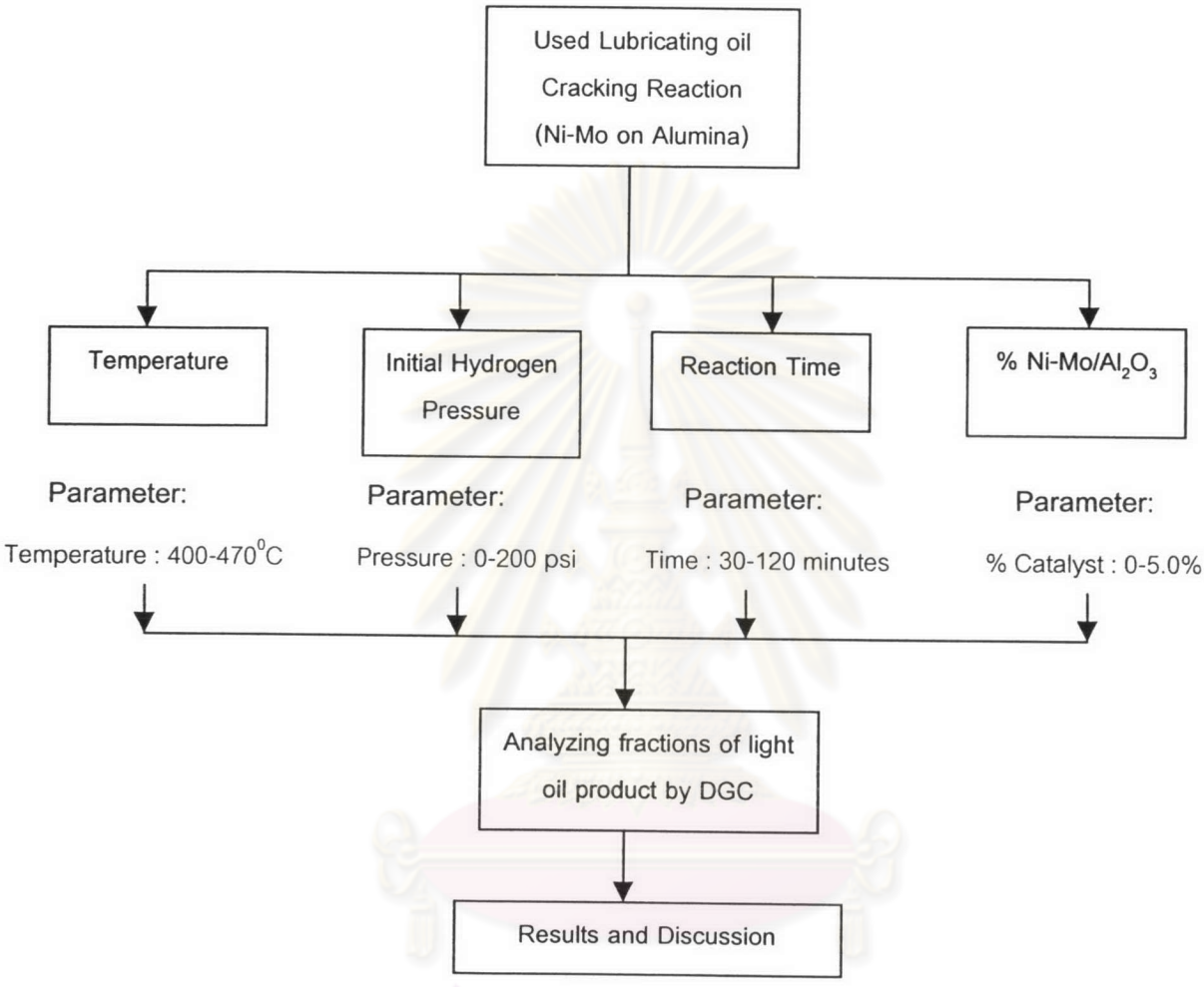

Figure 3.5 Experiment scheme hydrocracking of used lubricating oil with Ni-Mo on alumina.

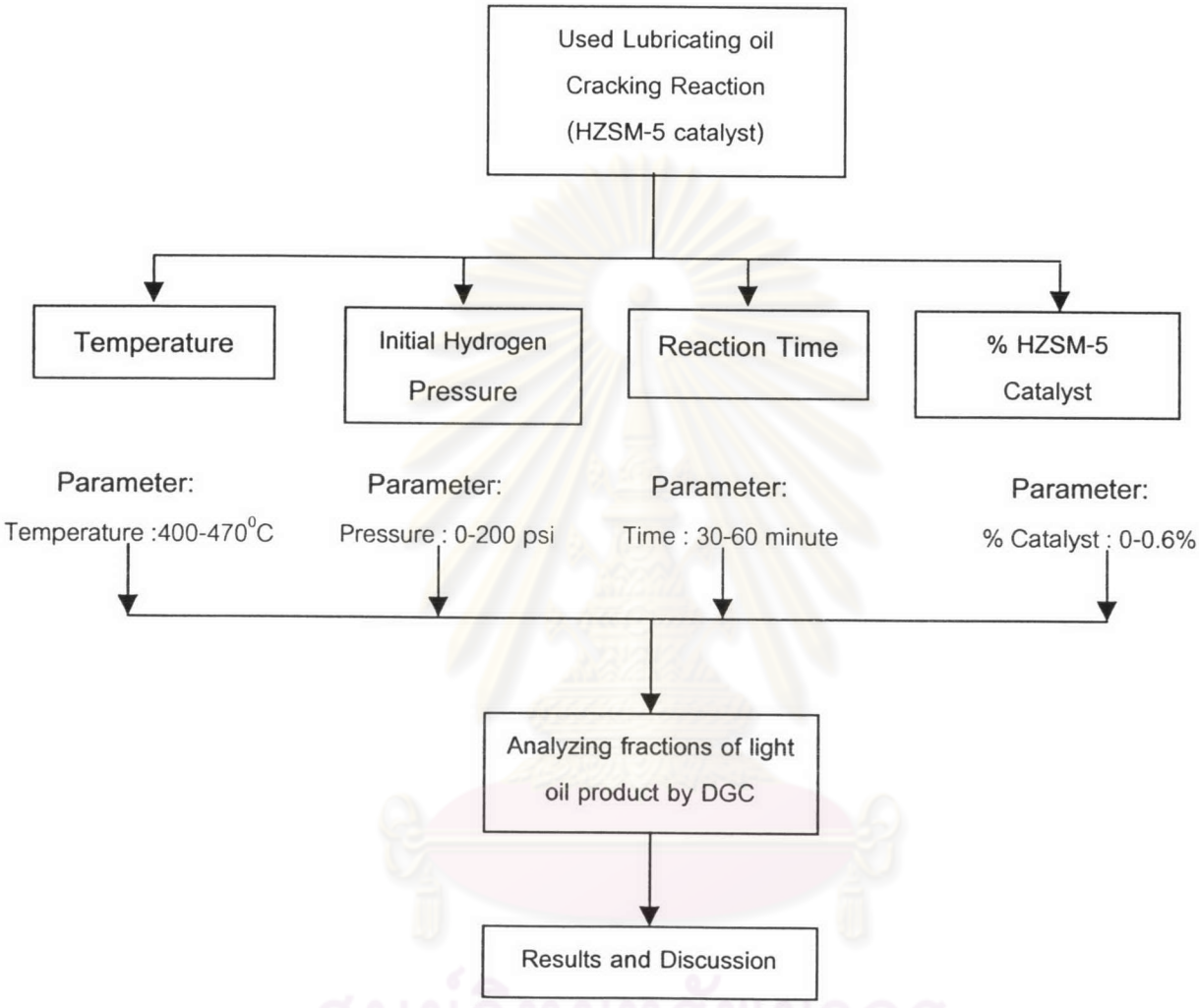

Figure 3.6 Experiment scheme of hydrocracking of used lubricating oil with HZSM-5 catalyst.

After the reaction, the product 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 catalyst and residue were separated from liquid products by vacuum filtration. The liquid products were analzed 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 metrod. Additional replications were performed on a random basis. After the entire set of reactions was completed, the reactions were replicated, since these reactions produced changes in the yield of the reactants and in the amout of gas.

After the Simulated Distillation recovery was determined, the weight percentages of liquid products boiling in the ranges ibp-100 (where ibp is initial boiling point), <200, <300, <400, and 500 ^{0}C were calculated. Standard deviations for these weight percentages were calculated by using standard deviations obtained in the product distributions and from Simulated Distillation recovery. The total product distribution of the reaction product was calculated by Simulated Distillation.