CHAPTER III
EXPERIMENTAL

3.1 Materials

Beta zeolite with Si/Al ratios of 10, 30 and 100, X zeolite and Y zeolite were supplied by Institut Français du Pétrole (IFP), France. ZSM-5 was purchased from Süd-Chemie Inc., Kentucky, USA.

3.2 Equipment

3.2.1 Mercury Analyzer

Mercury analyzer NIC SP-3D (Nippon Instruments Corporation, Japan) was used to analyze the mercury concentration in heavy naphtha. In an automatic system, the feed sample was first decomposed by heating and vaporizing followed by collection of mercury vapors in a collector containing gold amalgam. Then, the mercury atoms were released by heating the collector. The mercury atoms were detected in an absorption cell of a cold vapor atomic absorption spectrometer.

3.3 Methodology

3.3.1 Kinetic Study of Mercury Adsorption in Batch System

Beta zeolite with Si/Al ratios of 10, 30 and 100, X zeolite, Y zeolite, and ZSM-5 zeolite were used to study adsorption kinetic in heavy naphtha. 0.1 g of adsorbent was added into 20 ml of heavy naphtha in a 20 ml screw cap glass vial for each run. The glass vial was immersed in the water controlled at 50°C by a magnetic hot plate. The samples were taken from the glass vial by a syringe and diluted in *p*-xylene. Then, the samples were analyzed by mercury analyzer (NIC SP-3D). The schematic of the batch system is shown in Figure 3.1.

3.3.2 Isotherm Study of Mercury Adsorption and Adsorption Study of Hg^0 in Heavy Naphtha in Batch System

The experiment set up was similar to the batch system in section 3.3.1. The concentrations of mercury in heavy naphtha in isotherm study were 50, 100, 200, and 300 ppb, respectively. For Hg^0 adsorption study, 300 ppb of Hg^0 was prepared by spiking Hg^0 into heavy naphtha without mercury. For each run, 0.1 g of adsorbent was put into 20 ml of heavy naphtha in a 20 ml screw cap glass vial. Then, the glass vial was immersed in the water controlled at 50°C by a magnetic hot plate. The samples were taken from the glass vial by a syringe and diluted in *p*-xylene after 6 h. Then, the samples were analyzed by mercury analyzer (NIC SP-3D).

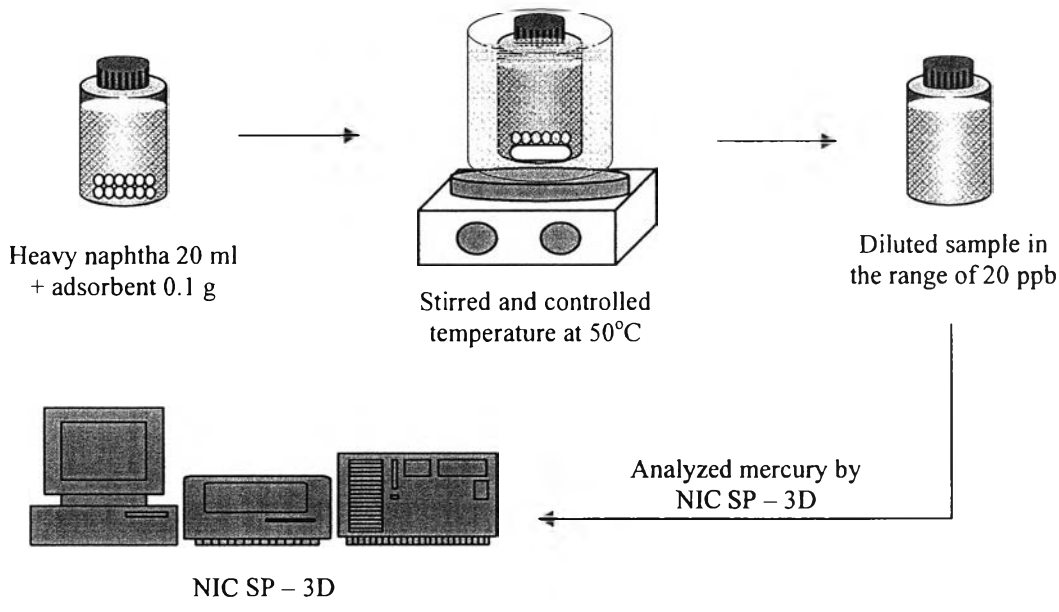

Figure 3.1 Schematic of the batch system.

3.3.3 Continuous System

Experiments were done in pilot plant unit 844 at Institut Français de Pétrole (IFP), Lyon, FRANCE. The schematic of continuous system is shown in Figure 3.2. The tests were performed in a reactor; the diameter and the height are 0.9 cm and 3.2 cm, respectively.

Before starting the experiment, the system was cleaned by flowing a large quantity of heavy naphtha to flush all impurities out. Then, 0.5 ml of adsorbent was loaded into the reactor. The adsorbent was mixed with SiC in order to maintain necessary bed height at approximately 3 cm.

After the adsorbent was loaded into the reactor, the reactor was heated to 200°C for 8 h to remove humidity and other impurities from the adsorbent. Then, the reactor was cooled to 30°C.

After the preparation step, feedstock (heavy naphtha) was fed into a feed drum and was pumped into the reactor by micrometric pump at a flow rate of 2 ml/min. The conditions of the reactor were set at 50°C and 7 bar. Sampling was carried out from the sample collecting drum and the effluent was sent into the waste disposal tank.

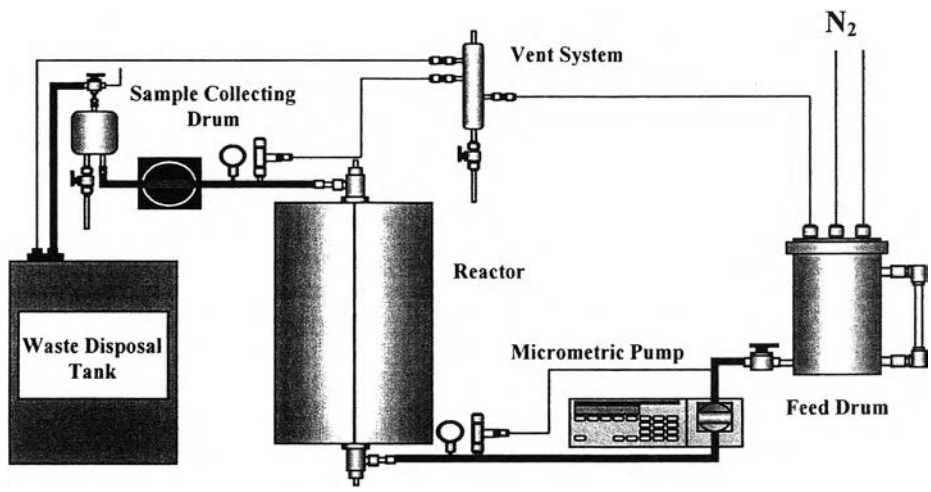

Figure 3.2 Process flow diagram of continuous system (Unit U844, IFP-LYON, FRANCE).