CHAPTER III
EXPERIMENTAL

3.1 Materials

The zeolite L (K-LTL) was produced by Tosoh Co. Zeolite Beta (NaH-BEA) and zeolite Omega (H-MAZ) were produced by Zeolyst International, USA. The commercial adsorbent CMG273 was obtained from The Aromatic Thailand Company Limited (ATC), Thailand and Institut Français du Pétrole (IFP), France. The properties of the Zeolites and CMG273 are mentioned in Table 3.1. Diphenylmercury (DPM) (99%) was purchased from ACROS ORGANICS, New Jersey, USA. n-Heptane (99.5%) was supplied by Lab-Scan, Thailand. Heavy naphtha and condensate were provided by The Aromatic Thailand Company Limited (ATC), Rayong, Thailand.

Table 3.1 Physical properties of Zeolites and commercial adsorbent CMG273

Adsorbent	Si/Al	Pore(Å)	Area (m^2/g)
Omega	2.4	7.4	-
Beta	8.3	7.4	660
L	2.8	7.1	300
CuS/Al_2O_3 (CMG273)	-	-	142

3.2 Experimental Equipment

3.2.1 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) was used to determine the phase transfer of adsorbents. The thermogravimetric analyzer (Du Pont TAG 2950, France) was programmed to heat up from room temperature to 900°C at the rate of 10°C/min. The mass change with temperature increment was monitored and recorded by TGA thermal analyser.

3.2.2 Mercury Analyzer

Analysis of mercury was performed by mercury analyzer NIC SP-3D (Nipon Instruments Corporation, Japan). The method based on the atomic absorption of 253.65 nm of mercury vapor. In an automatic system, the feed sample was first decomposed by heating and vaporizing then followed by collection of mercury vapors in a collector as gold amalgam and, then were set freed as mercury atoms by heating the mercury collector. The mercury atoms were, then, detected in a absorption cell of a cold vapor atomic absorption spectroscope.

3.3 Methodology

3.3.1 Preparation of Diphenylmercury in n-Heptane and Heavy Naphtha

Both n-heptane and heavy naphtha (mercury free) were spiked with diphenylmercury (DPM) at various concentrations by using equation 3.1 as follows:

$$Hg, mg/l = \frac{10^3 \times (0.565) \times M}{0.500} \qquad (3.1)$$

where

M	= weight of DPM, g
0.500	= volume of volumetric flask, liter
0.565	= molecular mass of mercury (200.593) divided by the molecular mass of DPM (354.798)
10^3	= factor to convert g to mg

Low concentration (2.0 mg/l) was prepared by weighing 0.0018 g of DPM to the nearest 0.1 mg and then transferred quantitatively with n-heptane into a 500 ml volumetric fiask. The volumetric flask was capped and inverted

several times for thorough mixing and finally, was transferred into a plastic bottle and was kept refrigerated at 4 °C for use within one week.

High concentration (5.0 mg/l) was prepared by weighing 0.0045 g of DPM to the nearest 0.1 mg and was diluted/mixed in the same manner as mentioned previously.

3.3.2 Stability of Mercury in Borosilicate Glass Vial

Suitable containers were required to eliminate evaporation and adsorption losses of mercury on the container's wall. Borosilicate glass vials (commonly available) of 2 ml capacity were tested by storing 2 ppm of DPM concentration for 7 days at 30°C in a temperature controlled bath. Samples were taken and analyzed by mercury analyzer (NIC SP-3D) at end of each day.

3.3.2 Kinetic Study of DPM Adsorption (Batch)

Zeolite L, Omega, Beta and CMG273 were used to study adsorption kinetics is a batch system using 2.0 mg/l and 5.0 mg/l of DPM in n-heptane. For each run, 0.1000 g of adsorbent was added into 15 ml of DPM solution in 30 ml screw cap plastic bottle (polypropylene type: stability was checked for DPM in n-heptane by Namprai, O., 2005). Bottles were place in a temperature controlled water bath at 30, 40 and 50°C. Samples were taken from the bottle each time by using syringe then diluted and stored for analysis. Samples were analyzed by Mercury Analyzer (NIC SP-3D) followed by UOP 938-00 method.

3.3.3 Isotherm Study of DPM Adsorption (Batch)

The adsorption isotherms were constructed in a batch system using DPM concentrations of 2.0, 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0 mg/l. In each run 0.1000 g of adsorbent was mixed with mercury contaminated solution in a 30 ml screw cap plastic bottle (polypropylene type: stability was checked for DPM in n-heptane by Nampri, O., 2005) and was sampled in a span of 6 hrs (according to the kinetics results of each adsorbents). Samples were analyzed by Mercury Analyzer (NIC SP-3D) followed by UOP 938-00 method.

3.3.4 Continuous System

Experiments were done in Pilot Plant Unit 844 at Institut Français de Pétole (IFP), Lyon, FRANCE. The schematic of continuous system is shown in Figure 3.1.

Before starting the experiment, the system was cleaned by flowing a large quantity of n-heptane to flush all impurities out from the system. Then 2.0 g of adsorbent was loaded into adsorber. The adsorbent was diluted with SiC in order to maintain necessary bed height.

After loading the adsorbent into the adsorber, the adsorber was heated to 200°C for 8 hrs to remove humidity and other impurities from the adsorbent. Then, the adsorber was cooled to 30°C.

After the preparation step, feedstock (heavy naphtha) containing DPM (2.0 ppm) was fed into a feed drum and was pumped into the adsorber by micrometric pump at a flow rate of 1.5 ml/min. The conditions of adsorber were set at 30°C and 7 bar. The sample after passing through the adsorbent was the sampled from sample collecting drum and the excess of effluent was sent into the waste disposal tank.

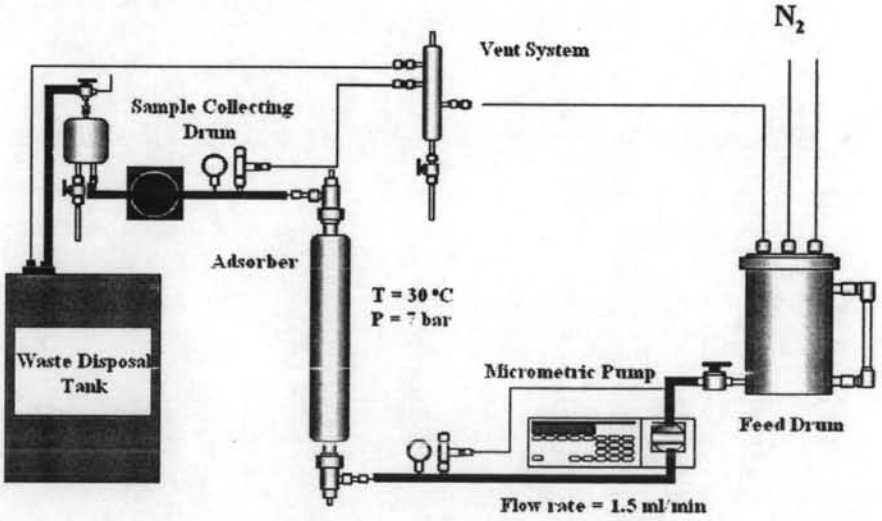

Figure 3.1 Process flow diagram of continuous system (Unit U844, IFP-LYON, FRANCE)

In each experiment, the complete consisted of adsorption and desorption. In the adsorption part, heavy naphtha spiked with 2 ppm of DPM

was used as feedstock for all adsorbents, while in the desorption pure n-heptane was used as a washing solution for cleaning the system. The unit was washed until the DPM concentration at the outlet reached to a minimum level.