

## CHAPTER III EXPERIMENTAL

### 3.1 Materials

#### 3.1.1 Chemicals

All chemicals used in this work are shown in Table 3.1.

**Table 3.1** Chemicals used in this work

Chemical	Source	Purity
n-Hexane (C <sub>6</sub> )	Aldrich	99.0 %
1-Hexene (1-C <sub>6</sub> )	Aldrich	97.0 %
n-Octane (C <sub>8</sub> )	Aldrich	99.5 %
1-Octene (1-C <sub>8</sub> )	Aldrich	98.0 %
n-Nonane (C <sub>9</sub> )	Aldrich	99.5 %
1-Nonene (1-C <sub>9</sub> )	Aldrich	97.0 %
n-Decane (C <sub>10</sub> )	Aldrich	99.0 %
1-Decene (1-C <sub>10</sub> )	Aldrich	94.0 %
n-Dodecane (C <sub>12</sub> )	Aldrich	99.0 %
1-Dodecene (1-C <sub>12</sub> )	Aldrich	95.0 %
n-Tetradecane (C <sub>14</sub> )	Aldrich	99.0 %
1-Tetradecene (1-C <sub>14</sub> )	Aldrich	92.0 %
n-Hexadecane (C <sub>16</sub> )	Aldrich	99.0 %
1-Hexadecene (1-C <sub>16</sub> )	Aldrich	92.0 %
n-Octadecane (C <sub>18</sub> )	Aldrich	99.0 %
1-Octadecene (1-C <sub>18</sub> )	Aldrich	90.0 %
n-Eicosane (C <sub>20</sub> )	Aldrich	99.0 %
1-Eicosene (1-C <sub>20</sub> )	Aldrich	90.0 %
iso-Octane	Aldrich	99.0 %
1,3,5-triethylbenzene	Aldrich	98.0 %

### 3.1.2 Adsorbents

Commercially available silicalite and NaX zeolites (1.04 % LOI and 4.60% LOI) with 20 % binder were provided by UOP LLC.

## 3.2 Experiment

### 3.2.1 Single Component Adsorption Experiment

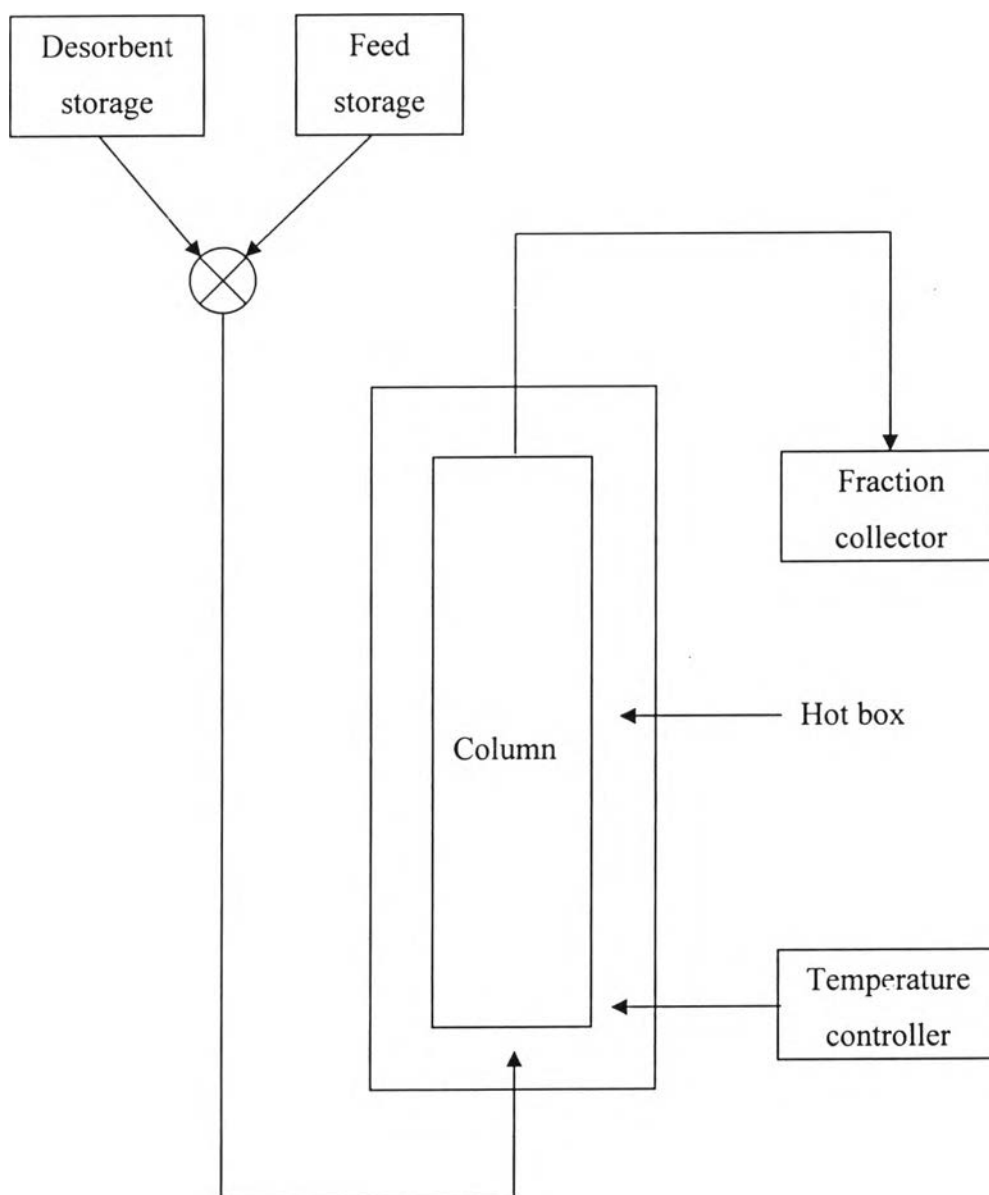
Equilibrium single component adsorption experiments of hexane, 1-hexene, octane, 1-octene, decane, 1-decene, dodecane, 1-dodecene, tetradecane, 1-tetradecene, hexadecane, 1-hexadecene, octadecane, 1-octadecene, eicosane and 1-eicosene on silicalite were performed. Silicalite was calcined at 673 K for 3 hours. Solutions of hydrocarbon in a non-adsorbed solvent, iso-octane, were prepared with difference concentrations. Approximately, one gram of silicalite was put into a vial. Then, about 10 ml of the solution was injected into the silicalite-containing vial and a blank vial without silicalite. After the solution was injected, the vial was immediately sealed and weighed to determine the solution composition. Samples were kept at room temperature ( $\sim 25$  °C) and shaken frequently until they reached equilibrium. Liquid samples were taken and analyzed by a Hewlett Packard 5890 series gas chromatograph (GC) with a flame ionization detector (FID).

### 3.2.2 Binary Component Competitive Adsorption Experiment

The same procedure described above was applied for equilibrium competitive adsorption experiments except the solution preparation. Mixtures of n-paraffin and n-olefin at the same carbon number (hexane/1-hexene, octane/1-octene, decane/1-decene, dodecane/1-dodecene, tetradecane/1-tetradecene, hexadecane/1-hexadecene, octadecane/1-octadecene, and eicosane/1-eicosene) were prepared with equimolar ratio in the non-adsorbed solvent.

### 3.2.3 Dynamic Adsorption: Multi-component Pulse Test

In this experiment, silicalite and NaX were used as the adsorbents and there was just only one composition of the feed. Figure 3.1 shows the experimental set-up for the multi-component pulse test unit. Adsorbent was packed in a 70 ml column with 0.75 and 158 cm as the inside diameter and length. Glass wood was placed at both ends of the column to block the adsorbent. The column was positioned in the controlled temperature hot box and connected to a pulse injection unit. The experiment was started with feeding the desorbent, which was a mixture between 40 % iso-octane and 60 % octane, to the column at a constant flow rate of 1.2 ml/min. After the column was saturated with the desorbent, a heater was turned on. A feed solution mixture consisted of 5.26 wt% of the following hydrocarbons: hexane, 1-hexene, octane, 1-octene, nonane, 1-nonene, decane, 1-decene, dodecane, 1-dodecene, tetradecane, 1-tetradecene, hexadecane, 1-hexadecene, octadecane, 1-octadecene, eicosane, 1-eicosene and 1,3,5-triethylbenzene. After reaching the desired temperature, 120 °C, about 5 ml of the feed was injected into the desorbent stream, which continuously flowed through the column, through the two-way valve. At the same time, the fraction collector was started to collect the system effluent every 2 min. Each fraction, approximately 2.4 ml of the sample, was analyzed by a Hewlett Packard 5890 series II gas chromatograph equipped with a FID. The concentration of all components was plotted versus time or volume and then the n-paraffin and n-olefin selectivity was calculated.



**Figure 3.1** Experimental set-up of the pulse test unit.