CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Chemicals

- *m*-chloronitrobenzene 98.0% purity, Acros
- *p*-chloronitrobenzene 99.5% purity , Acros

3.1.2 Adsorbents

Alkali and alkali earth ion-exchanged faujasite zeolites from UOP, A Honeywell Company, USA with chemical compositions are shown in Table 3.1.

Table 3.1 Chemical composition for various faujasite zeolite

Sample	Chemical composition
NaX	$Na_{84}(AlO_2)_{84}(SiO_2)_{108}$
MgX	$Mg_{30}Na_{24}(AlO_2)_{84}(SiO_2)_{108}$
CaX	$Ca_{40}Na_4(AlO_2)_{84}(SiO_2)_{108}$
SrX	$Sr_{41}Na_2(AlO_2)_{84}(SiO_2)_{108}$
BaX	$Ba_{41}Na_2(AlO_2)_{84}(SiO_2)_{108}$
NaY	$Na_{53}(AlO_2)_{53}(SiO_2)_{139}$
MgY	$Mg_{20}Na_{13}(AlO_2)_{53}(SiO_2)_{139}$
CaY	$Ca_{23}Na_7(AlO_2)_{53}(SiO_2)_{139}$
SrY	$Sr_{24}Na_5(AlO_2)_{53}(SiO_2)_{139}$
BaY	$Ba_{24}Na_5(AlO_2)_{53}(SiO_2)_{139}$

3.1.3 Solvents

- n-haxane 99.0% purity, Burdick & Jackson
- Dodecane 97.0% purity, Fluka

3.2 Equipment

- Gas chromatograph (GC) equipped with a SUPELCOWAXTH capillary column and an FID detector
- Cooling water pump
- Crystallization unit
- Controlled temperature shaker

3.3 Methodology

3.3.1 Static Adsorption Study of *m*- and *p*-CNB

3.3.1.1 Single Component Adsorption Study

Equilibrium single component adsorption experiments of *m*-CNB and *p*-CNB on MgX, CaX, SrX, BaX, MgY, CaY, SrY, and BaY were conducted. All zeolites were pretreated by calcination at the temperature of 350°C for an hour. *m*-CNB and *p*-CNB were prepared with different concentrations between 1–8 wt%. Hexane was used as a solvent and it was expected not to adsorb on X and Y zeolites in the presence of the CNBs. The total weight of the solution was 2.6 g, which contained 7 wt% dodecane used as a tracer. 0.22 g of zeolite was added in the solutions. The vials were sealed to prevent evaporation and then put into the shaker controlled temperature at 30°C for 24 hours. The liquid samples were taken and analyzed by a GC for measuring the residual concentration of CNBs in the solution at the equilibrium condition. Finally, the adsorption capacity of both CNBs was determined.

3.2.1.2 Binary Component Adsorption Study

The same procedure described above was applied for equilibrium competitive adsorption experiments except the solution preparation. Mixtures of *m*-CNB and *p*-CNB were prepared in an *m*-CNB/ *p*-CNB molar ratio of 1:1 for any concentration to study the interaction of each isomer on the adsorption.

3.3.2 Effect of FAU Zeolite on Crystallization Study of *m*- and *p*-CNB

The crystallization unit was shown in Figure 3.1. The 7 g of solid mixture, 65 wt% *m*-CNB and 35 wt% *p*-CNB, was melted to obtain a homogeneous solution. The zeolites were calcined at 350°C for an hour before the experiments. Ten grains of the employed zeolite were added into the CNB mixture. The system was cooled by the cooling water to 22°C, the eutectic temperature of the binary *m*- and *p*-CNB system. Then, both mixed precipitates and mother liquor were collected, washed, and dissolved with hexane for the CNB composition analysis by the GC. Note that this experiment must be done in a clean area, avoiding dust contaminants in the system that may potentially act as a seed for crystallization.

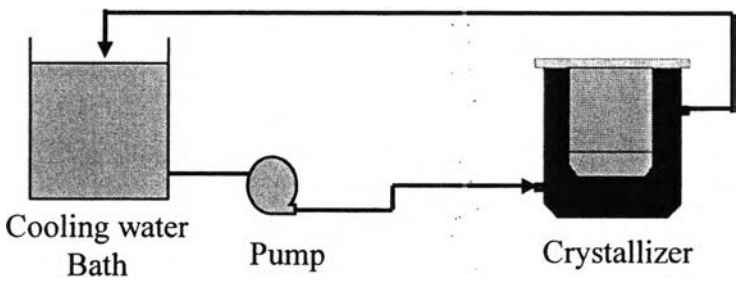

Figure 3.1 Crystallization unit.