CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Chemicals and Solvent

- *m*-chloronitrobenzene 99.0% purity, Italmar
- *p*-chloronitrobenzene 99.5% purity , Italmar
- *n*-haxane 99.0% purity, Acros
- *n*-dodecane 99.0% purity, Merck

3.1.2 Adsorbents

- Alkali and alkali earth ion-exchanged faujasite zeolites were obtained from UOP, A Honeywell Company, USA. The chemical compositions are shown below:

  NaX ($Na_{84}(AlO_2)_{84}(SiO_2)_{108}$)
  CaX ($Ca_{40}Na_4(AlO_2)_{84}(SiO_2)_{108}$)
  BaX ($Ba_{41}Na_2(AlO_2)_{84}(SiO_2)_{108}$)
  NaY ($Na_{53}(AlO_2)_{53}(SiO_2)_{139}$)
  CaY ($Ca_{23}Na_7(AlO_2)_{53}(SiO_2)_{139}$)
  KY ($K_{51}Na_2(AlO_2)_{53}(SiO_2)_{139}$)
- SiO_2, Tokusil
- Al_2O_3, Saint-Gobain NorPro
- Activated carbon, Sigma aldrich
- Glass bead

3.2 Equipment

- A gas chromatograph (GC) equipped with a SUPELCOWAXTH capillary column and a flame ionization detector (FID)
- A cooling water bath
- A crystallization unit

- A heater
- An insulated chamber

3.3 Methodology

3.3.1 Selectivity and Adsorption Capacities of *m*-CNB and *p*-CNB

Equilibrium binary component adsorption experiments of *m*-CNB and *p*-CNB on NaX, CaX, BaX, NaY, CaY, KY, Al_2O_3, SiO_2, activated carbon and glass beads were performed. Al_2O_3, SiO_2, activated carbon and glass beads were pelletized, mashed and sieved to the particle size 20/40 mesh. All adsorbents were calcined at 350 °C for 1 h. Hexane was used as the solvent in all experiments as it does not adsorb on the adsorbents in the presence of aromatics. Solution of CNBs was prepared by 2% *m*-CNB and 2% *p*-CNB in *n*-hexane. Then, 12 g of solution containing 7 wt% dodecane as a tracer was added to 1 g of an adsorbent containing vial and a blank vial without an adsorbent. The vial was then sealed to prevent evaporation. Samples were kept at 30 °C and shaken frequently with a constant temperature shaker until they reached equilibrium, about 24 h. Liquid samples were taken and analyzed by a Hewlett Packard 6890 series gas chromatograph (GC) with a flame ionization detector (FID) and the SUPELCOWAXTH 10 capillary column, which can separate all isomers of CNB from each other to determine the adsorption capacity.

3.3.2 Effects of Feed Composition on *m*- and *p*-CNB Crystallization

To study the effects of the feed composition of crystallization of *m*- and *p*-CNB, the feed solutions were prepared with different *m*-CNB compositions consisting of below the eutectic, at the eutectic, and above the eutectic composition (61.0, 62.9, and 63.5 wt% of *m*-CNB in the feed). Seven grams of *m*- and *p*-CNB solid mixture were melted in the crystallizer to obtain a homogeneous solution. Then, the system was cooled by the cooling water from 30 °C to the crystallization temperature, at which the crystal was initially formed. The crystals were collected, washed, and dissolved with hexane. The dissolved crystals were analyzed for the CNB composition by the GC.

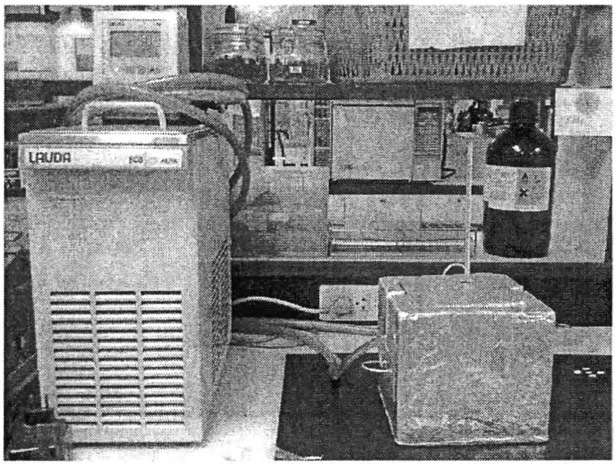

Figure 3.1 Crystallization unit.

3.3.3 Effects of Adsorbents on the Crystallization of *m*- and *p*-CNB

The influence of adsorbents on the crystallization was studied by using the starting composition at and above the eutectic composition (62.9 and 63.5 wt% of *m*-CNB in the feed) and various adsorbents (NaX, CaX, BaX, NaY, CaY, KY, Al_2O_3, SiO_2, activated carbon and glass bead). The CNB liquid mixture was prepared with the same procedure as in Part 3.3.2. Adsorbents were calcined at 350°C for an hour before the experiment. Five grains of adsorbents were added at the center of the CNB mixture in the crystallizer. The mixture was stirred to minimize any concentration gradient in the solution and then collected to check the composition after adding adsorbents by the GC. The system was cooled by cooling water at the cooling rate of 1°C/hr to the crystallization temperature, at which the crystal was initially formed. The crystals from 8 positions in two areas, area (a) and area (b), as shown in Figure 3.2, were collected, washed and dissolved with hexane. The dissolved crystals were measured for the CNB compositions by the GC. The experiment was repeated by using 10 grains of each adsorbent.

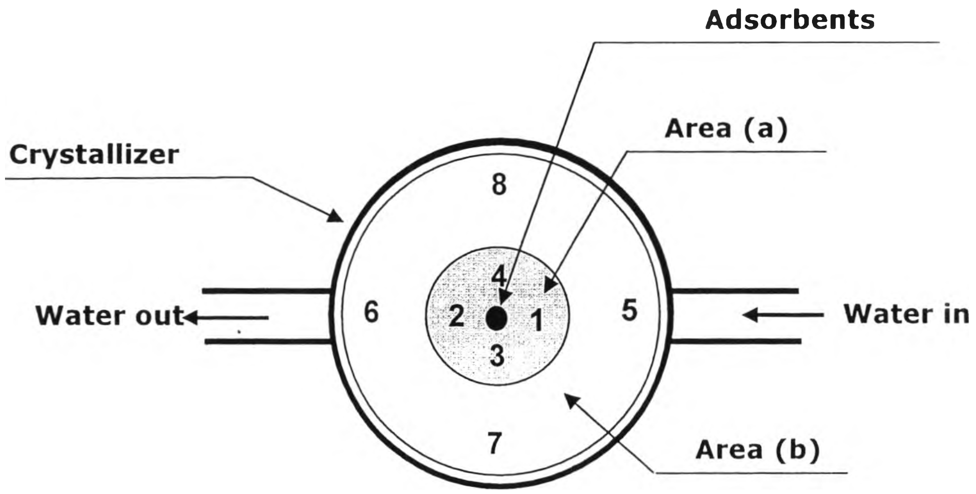

Figure 3.2 Locations where crystals were collected for studying effect of the number of adsorbents on the crystallization.