

CHAPTER III EXPERIMENTAL

3.1 Materials and Equipment

3.1.1 Equipment

- Gas chromatograph (GC) equipped with a SUPELCOWAXTH capillary column and an FID detector
- Cooling water pump
- Heater
- Water bath
- Crystallizer

3.1.2 Chemicals

- *m*-chloronitrobenzene 99.0% purity, Acros
- *p*-chloronitrobenzene 99.5% purity, Acros

3.1.3 Solvents

- n-hexane 99.5 % purity, Acros

3.1.4 Adsorbents

- Alkali and alkaline earth ion-exchanged faujasite zeolite from UOP, a Honeywell Company, USA.
- Chemical composition :
 - BaX(Ba₄₁Na₂(AlO₂)₈₄(SiO₂)₁₀₈)
 - KY(K₅₁Na₂(AlO₂)₅₃(SiO₂)₁₃₉)
 - NaX(Na₈₄(AlO₂)₈₄(SiO₂)₁₀₈)
 - Activated carbon, Sigma aldrich
 - Silica gel (SiO₂), Tokusil

3.2 Experimental Procedures

3.2.1 Effect of Feed Composition on *m*- and *p*-CNB Crystallization

Seven grams of a solid mixture, *m*- and *p*-CNB, were melted to obtain a homogeneous solution with different *m*-CNB concentrations, below the eutectic, at the eutectic, and above the eutectic composition. The liquid mixture was measured for the CNB composition by use the GC equipped with a SUPELCOWAXTM capillary column and an FID detector. The CNB mixture was added in the crystallizer that was set as shown in Figure 3.1. The system was cooled by cooling water at the cooling rate of 1°C/hr to a crystallization temperature, where the precipitates initially formed. All precipitates were collected from the crystallizer, washed, and dissolved with hexane. The dissolved crystals were measured for the CNB composition by use the GC.

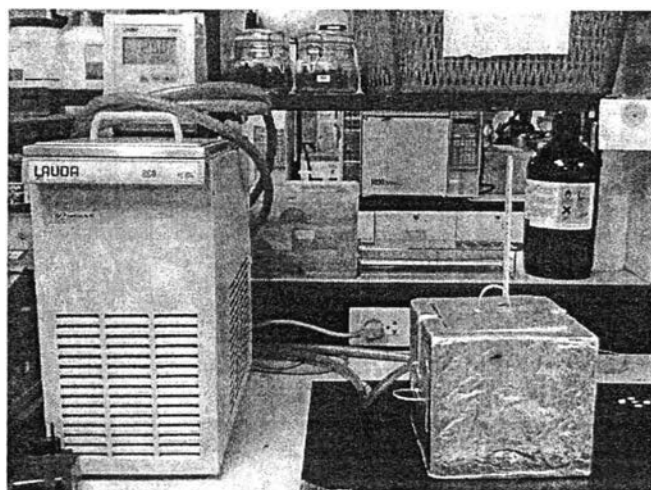


Figure 3.1 Crystallization unit.

3.2.2 Effect of Zeolite on *m*- and *p*-CNB Crystallization

A phase diagram study with the presence of KY zeolite on the crystallization was studied by varying starting compositions, below the eutectic, at the eutectic, and above the eutectic composition, and types of adsorbents (NaX, BaX, KY, Activated carbon, and Silica gel were pelletized, mashed and sieved to the particle size 20/40 mesh). The CNB liquid mixture was prepared with the same procedure as 3.2.1. Zeolites were calcined at 350°C for an hour before the experiment. Five grains of KY zeolites were added at the center of CNBs mixture in the crystallizer. The mixture was stirred and collected to check the composition after adding zeolites by using the GC. The system was cooled by cooling water at the cooling rate of 1°C/hr to crystallization temperature where the precipitates initially formed. The precipitates 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 use the GC. The experiments were repeated by changing the zeolite.

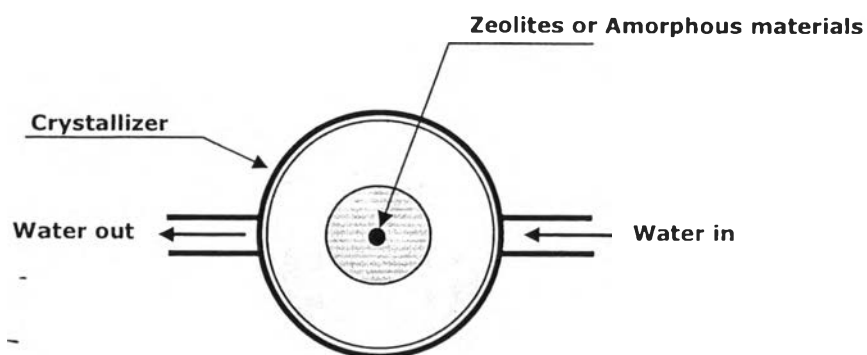


Figure 3.2 Locations where solids are collected to study this effect on the crystallization