



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*-hexane 99.0% purity, Acros

3.1.2 Adsorbents

Alkali and alkali earth ion-exchanged faujasite zeolites and silicalite from UOP, A Honeywell Company, USA with chemical compositions are shown as follows:

- NaX ($\text{Na}_{84}(\text{AlO}_2)_{84}(\text{SiO}_2)_{108}$)
- CaX ($\text{Ca}_{40}\text{Na}_4(\text{AlO}_2)_{84}(\text{SiO}_2)_{108}$)
- BaX ($\text{Ba}_{41}\text{Na}_2(\text{AlO}_2)_{84}(\text{SiO}_2)_{108}$)
- NaY ($\text{Na}_{53}(\text{AlO}_2)_{53}(\text{SiO}_2)_{139}$)
- CaY ($\text{Ca}_{23}\text{Na}_7(\text{AlO}_2)_{53}(\text{SiO}_2)_{139}$)
- Silicalite
- Granular activated carbon, Sigma-aldrich

3.2 Equipment

- A gas chromatograph (GC) equipped with a SUPELCOWAXTH capillary column and a flame ionization detector (FID)
- A crystallization unit
- A heater
- An insulated chamber

3.3 Methodology

3.3.1 Effect of feed composition on *m*- and *p*-CNB crystallization

The crystallization unit is shown in Figure 3.1. The feed solutions were prepared with a different *m*-CNB composition (61.0, 62.9, and 65.0 wt% of *m*-CNB in 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 precipitate was initially formed. The mixed precipitates and mother liquor were collected, washed, and dissolved with hexane. The composition of *m*- and *p*-CNB in the feed, mixed precipitates and mother liquor were analyzed by a GC equipped with a SUPELCOWAXTH capillary column and a flame ionization detector (FID).

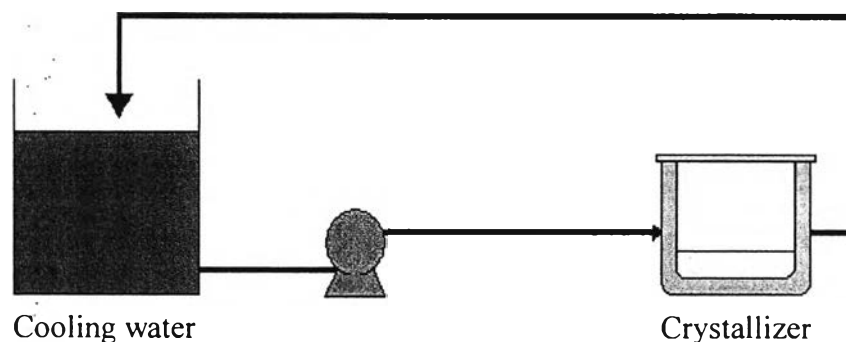


Figure 3.1 Crystallization unit.

3.3.2 Effect of FAU zeolite on *m*- and *p*-CNB crystallization

The effect of FAU zeolite on *m*- and *p*-CNB crystallization was investigated by adding NaX, CaX, BaX, NaY, and CaY in the feed having the same composition with above. All zeolites were calcined at 350°C for an hour before the experiment. A number of the zeolite was added at the center of the feed. The mixture was stirred to minimize any concentration gradient in the solution and then collected to check the composition after adding zeolite. The same procedure described in 3.3.1 was then repeated. To study the tendency of CNB precipitate composition in the

condition with a zeolite, the precipitates located at the positions 1 to 8 in two areas, area (a) and area (b), as shown in Figure 3.2, were collected. Moreover, in order to ensure that the system can be reproduced, the mixture was heated backwards to the homogeneous liquid phase and then recrystallized.

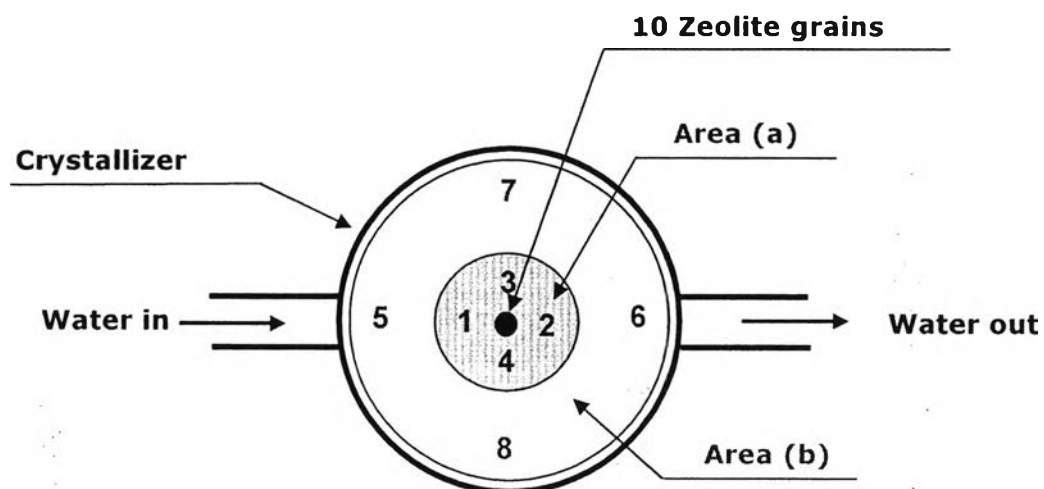


Figure 3.2 Locations where precipitates are collected for *m*- and *p*-CNB composition analysis.

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.