



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 from UOP, A Honeywell Company, USA with chemical compositions are shown below

- 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 ($\text{Ca}_{23}\text{Na}_7(\text{AlO}_2)_{53}(\text{SiO}_2)_{139}$)

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 Effects 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 compositions (61.0, 62.9, and 65.0 wt% of *m*-CNB). The liquid mixture was measured for the CNB compositions by using the GC equipped with a SUPELCOWAXTH 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 crystallizer, washed and dissolved with hexane. The dissolved crystals were measured the CNB composition by using the GC.

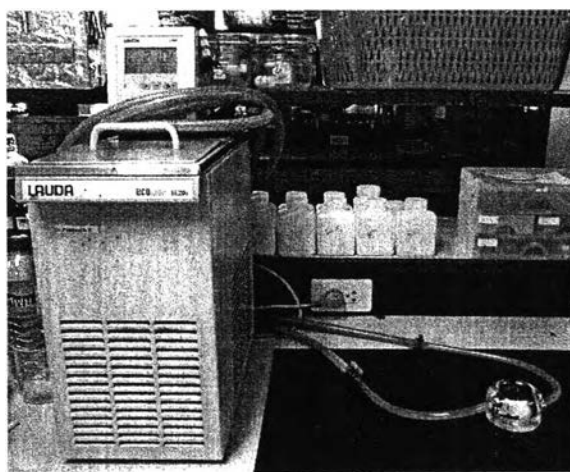


Figure 3.1 Crystallization unit.

3.3.2 Effects of FAU Zeolites on *m*- and *p*-CNB Crystallization

The influence of the number of FAU zeolites on the crystallization was studied by varying starting compositions (61.0 and 65.0 wt% of *m*-CNB in the feed) and types of adsorbents (NaX, CaX, BaX, NaY and CaY). The CNB liquid mixture was prepared the same procedure as 3.3.1. Zeolites were calcined at 350°C for an hour before the experiment. Five grains of a zeolite were added at the center of

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 the zeolite by using the GC. The system was cooled by cooling water at the cooling rate of 1°C/hr to a crystallization temperature where the precipitates initially formed. The precipitates 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 using the GC. The experiment was repeated by using 10 grains of zeolite.

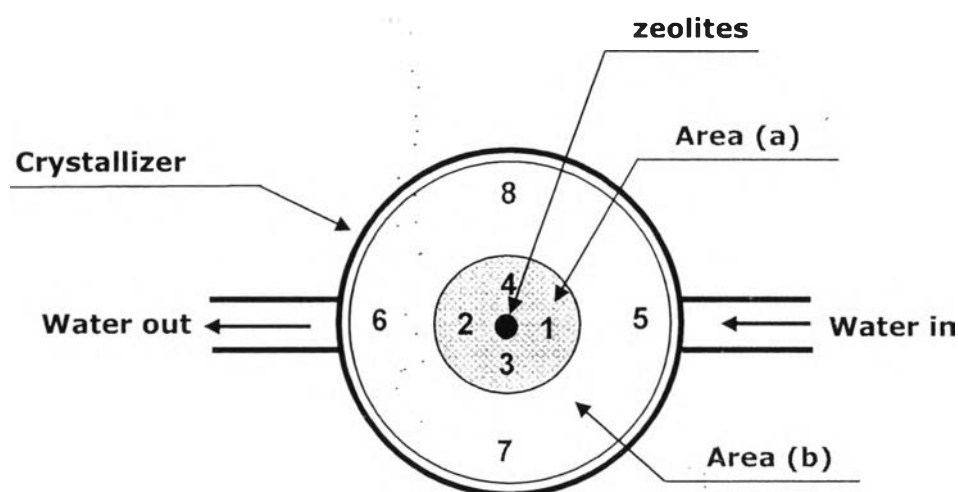


Figure 3.2 Locations where precipitates were collected for studying effect of the number of FAU zeolites on the crystallization.

3.3.3 Influences of Seeds on the Crystallization

The experiment was carried out as described in 3.3.2, but seeds were added in the CNB mixture instead of a zeolite at 24°C. Two types of crystal were used as the seed, *m*- and *p*-CNB. *m*-CNB seed and *p*-CNB seed were added in the CNB mixture as shown in Figure 3.3 (a) and Figure 3.3 (b), respectively. In addition, both *m*- and *p*-CNB seeds were placed in the CNB mixture as shown in Figure 3.3 (c)

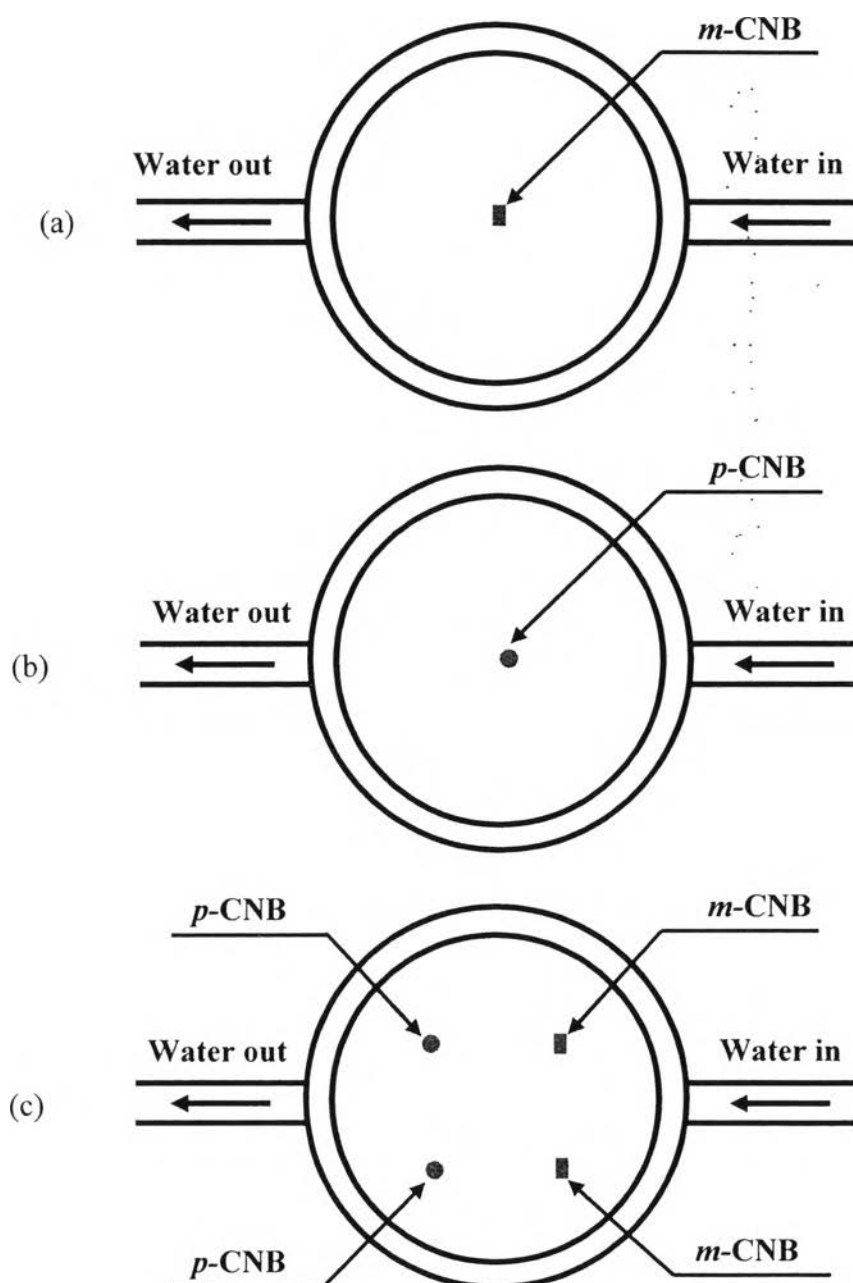


Figure 3.3 Types and locations of seed crystals in the crystallizer (a) *m*-CNB seed, (b) *p*-CNB seed and (c) *m*-CNB seeds and *p*-CNB seeds.