

CHAPTER III

METHODOLOGY

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 :



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 the GC equipped with a SUPELCOWAXTH capillary column and an FID detector. The CNB mixture was added in the crystallizer that is 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 form. All precipitates were collected from the crystallizer, washed, and dissolved with hexane. The dissolved crystals were measured for the CNB composition by the GC.

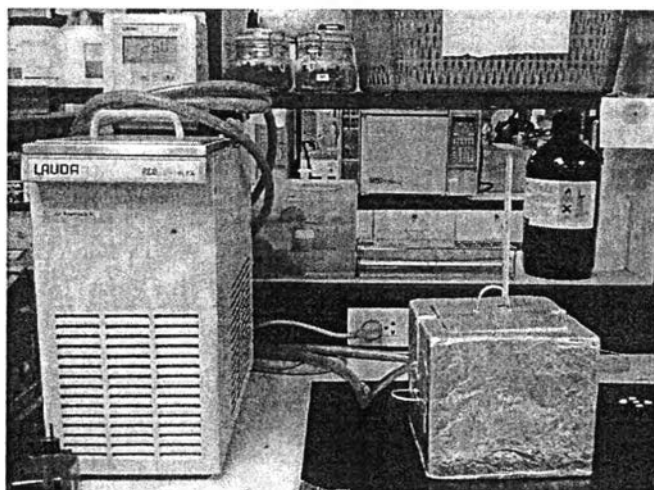


Figure 3.1 Crystallization Unit.

3.2.2 Effect of Particle Size 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 various particle size of purity and impurity substance (*m*-, *p*-CNB and KY were pelletized, mashed and sieved to different the particle size). The CNBs liquid mixture was prepared the same procedure as 3.2.1. Zeolites were calcined at 350°C for an hour before the experiment. Five grains of the same size of KY zeolites or seed substances 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 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 were collected, as shown in Figure 3.2, washed, and dissolved with hexane. The dissolved crystals were measured for the CNB compositions by the GC. The experiment was repeated by varying the different particle size.

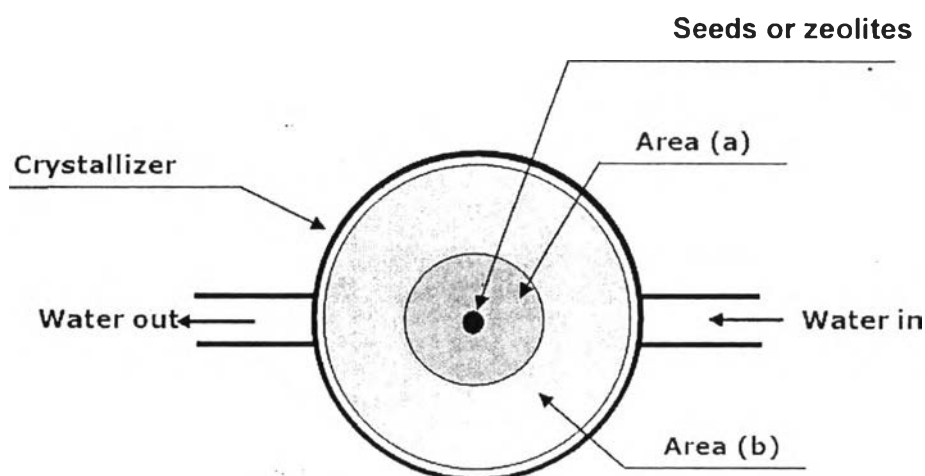


Figure 3.2 Locations where crystals are collected for studying effect of the type of seeds on the crystallization.