



CHAPTER III EXPERIMENTAL

3.1 Material and equipment

Equipment

A gas chromatograph (GC) equipped with an FID detector and a SUPEL-COWAXTH 10 capillary column.

Chemical

All chemicals used in this work are shown in Table 3.1.

Table 3.1 Chemicals used in this work

Chemicals	Source	Purity
<i>m</i> -Chloronitrobenzene	Italmar	99.0 %
<i>p</i> -Chloronitrobenzene	Italmar	99.5 %
n-Dodecane (C ₁₂)	Fluka	97.0 %
n-Hexane (C ₆) HPLC grade	Burdick & Jackson	99.0 %
Toluene	Carlo Erba	99.5 %
Benzene	Fisher Scientific	99.0 %
Nitrobenzene	Merck	99.0 %
<i>o</i> -Xylene	Sigma Aldrich	99.0 %
<i>o</i> -Dichlorobenzene	Carlo Erba	95.0 %

Adsorbent

The cation exchanged faujasite zeolites for X zeolites (Si/Al = 1.25) and Y zeolites (Si/Al = 2.5) were obtained from UOP LLC (Chemical compositions are shown in Table 3.2).

Table 3.2 Chemical composition for various faujasite zeolite

Sample	Chemical composition
LiX	$\text{Li}_{20}\text{Na}_{64}(\text{AlO}_2)_{84}(\text{SiO}_2)_{108}$
NaX	$\text{Na}_{84}(\text{AlO}_2)_{84}(\text{SiO}_2)_{108}$
KX	$\text{K}_{82}\text{Na}_2(\text{AlO}_2)_{84}(\text{SiO}_2)_{108}$
RbX	$\text{Rb}_{71}\text{Na}_{13}(\text{AlO}_2)_{84}(\text{SiO}_2)_{108}$
CsX	$\text{Cs}_{73}\text{Na}_{11}(\text{AlO}_2)_{84}(\text{SiO}_2)_{108}$
LiY	$\text{Li}_{22}\text{Na}_{31}(\text{AlO}_2)_{53}(\text{SiO}_2)_{139}$
NaY	$\text{Na}_{53}(\text{AlO}_2)_{53}(\text{SiO}_2)_{139}$
KY	$\text{K}_{51}\text{Na}_2(\text{AlO}_2)_{53}(\text{SiO}_2)_{139}$
RbY	$\text{Rb}_{47}\text{Na}_6(\text{AlO}_2)_{53}(\text{SiO}_2)_{139}$
CsY	$\text{Cs}_{49}\text{Na}_4(\text{AlO}_2)_{53}(\text{SiO}_2)_{139}$

3.2 Methodology

3.2.1 Single component adsorption experiment

Equilibrium single component adsorption experiments of *m*-CNB and *p*-CNB on LiX, NaX, KX, RbX, CsX, LiY, NaY, KY, RbY, and CsY were performed. All cationic exchanged faujasite were calcined at 350°C for 2 hours. *n*-Hexane was used as the solvent in all the experiments as it was expected not to adsorb on X and Y-type of zeolites in the presence of aromatics. Solution of CNBs was prepared with different concentrations from 1 to 8 wt%. Higher concentrations cannot be achieved due to the low solubility of CNBs. Approximately, 0.22 g of an adsorbent with a feed to adsorbent ratio equal to 12 was put into a vial. Then, 2.6 g of

solution contains 7 wt% of dodecane as a tracer was injected into the adsorbent containing vial and a blank vial without adsorbent. After the solution was injected, the vial was sealed to prevent evaporation. Samples were kept at 30°C and shaken frequently with a constant temperature shaker until they reached equilibrium. Liquid samples were taken and analyzed by a Hewlett Packard 6890 series gas chromatography (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.

3.2.2 Binary component competitive adsorption experiment

The same procedure described above was applied for equilibrium competitive adsorption experiments except the solution preparation. Mixtures of *m*-CNB and *p*-CNB were prepared in the non-adsorbed solvent to study the interaction of each isomer on the adsorption.

3.2.3 Effect of desorbent experiments

After choosing the appropriate adsorbent from single adsorption and binary adsorption, five desorbents: benzene, toluene, *o*-xylene, *p*-dichloro-benzene and nitrobenzene were chosen in this study. Mixtures of *m*-CNB, *p*-CNB and desorbent with an equimolar ratio were prepared to study the effect of desorbent to adsorbate and desorbent to adsorbent as well.

3.2.4 Effect of water content in the zeolite structure experiment

NaY, which was calcined at 350°C for 2 hours to eliminate physical adsorbed water and stored in a dessicator, was used directly as the dry sample. The wet sample, which was employed for investigating the influence of the water content on the adsorption behavior of the appropriate desorbent, *m*-CNB and *p*-CNB to the adsorbent, were prepared by equilibrating the dry sample with water vapor at an ambient temperature. The water contents of the adsorbent were 4.09%, 5.05% and 9.82% LOI (Loss of ignition). Water contents are calculated from conventional way by heating to 900°C. Weight loss is adsorbed water and organic component in the zeolite structure.