# CHAPTER III

# MATERIALS AND METHOD

## 3.1 Materials

Fly Ash is collected from the pulping process in commercial plantation area in Prachinburi province, Thailand.

## 3.2 Procedure for zeolite synthesis

### 3.2.1 Effect of NaOH/Fly ash weight ratio

1. Mix 10 g of fly ash with 17.5 g of NaOH (to make the NaOH / PFA of 1.75)
2. Burn the mixture in air at 550°C for 60 min using ashing furnace
3. Crush and dissolve the product in 85 mL of distilled water
4. Mix in a shaking water bath at 30 °C for12 h
5. Crystallize under static condition of 90 °C for 2 h
6. Collect the crystal (by filtration), wash several times with distilled water (until the solution reaches pH 10) and then dry overnight at 105 °C
7. Repeat Steps 1-6 with NaOH/Fly ash weight ratio 0, 0.5, 1, 1.25, 1.5, 2, 2.25, 2.5 and 3

### 3.2.2 Effect of Si/Al ratio and fusion temperature, time and crystallization temperature, time

1. Mix 10 g of fly ash (Si/Al mol ratio 2.29) with 17.5 g of NaOH (to make the NaOH / PFA of 1.75)
2. Burn the mixture in air at 550 °C for 60 min using ashing furnace
3. Crush and dissolve the product in 85 mL of distilled water
4. Mix in a shaking water bath at 30 °C for12 h
5. Crystallize under static condition of 90 °C for 2 h
6. Collect the crystal (by filtration), wash several times with distilled water (until the solution reaches pH 10) and then dry overnight at 105 °C
7. Repeat Steps 1-6 with fly ash that contain Si/Al mol ratio 4.05, 4.62
8. Repeat Steps 1-6 with fusion temperature 250, 350 and 450 °C

9. Repeat Steps 1-6 with fusion time 15, 30 and 45 min
10. Repeat Steps 1-6 with crystallization temperature 30 and 60 °C
11. Repeat Steps 1-6 with crystallization time 1 and 3 h

### 3.2.3 Effect of Amount of water and mixing temperature and time

1. Mix 10 g of fly ash (Si/Al mol ratio 2.32) with 17.5 g of NaOH (to make the NaOH / PFA of 1.75)
2. Burn the mixture in air at 550 °C for 60 min using ashing furnace
3. Crush and dissolve the product in 85 mL of distilled water
4. Mix in a shaking water bath at 30 °C for12 h
5. Crystallize under static condition of 90 °C for 2 h
6. Collect the crystal (by filtration), wash several times with distilled water (until the solution reaches pH 10) and then dry overnight at 105 °C
7. Repeat Steps 1-6 with amount of water 65 and 115 mL
8. Repeat Steps 1-6 with mixing temperature 60 and 90 °C
9. Repeat Steps 1-6 with mixing time 6, 24 and 48 h

## 3.3 Analytical Method and calculation of Cations Exchange Capacity

### 3.3.1 Analytical Method of Cations Exchange Capacity

1. Weigh 3.2 g of fly ash (quantity "$m$" in Eq. 3.1)
2. Add 27 mL of 1.0 N $CH_3COONa$ solution, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear
3. Decant the liquid, and repeat Step 2 two more times
4. Add 27 mL, of 99 % isopropyl alcohol, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear
5. Repeat the procedure described in Step 4 one more time
6. Add 27 mL of $CH_3COONH_4$ solution, stopper the tube, shake it in a mechanical shake for 5 min, and centrifuge it until the supernatant liquid is clear. Decant the washing solution into a 100 mL volumetric flash
7. Repeat the procedure as described in Step 6 one more time
8. Dilute the combined washing solution to 100 mL(quantity "$V$" in Eq. 3.1)) with ammonium acetate solution and determine the sodium concentration by atomic absorption emission spectroscopy (This leads to the quantity "$c_0 - c_b$" in Eq. 3.1).

### 3.3.2 Calculation of cation exchange capacity (CEC)

The sodium binding capacity is calculated from:

$$CEC = \frac{(c_0 - c_b)}{MW \times m} \times V \qquad (3.1)$$

Where $C_0$ is sodium concentration ($mgL^{-1}$), $C_b$ sodium concentration in blank ($mgL^{-1}$), $V$ the volume of the aqueous phase (mL), and $m$ the amount of zeolite (g), MW the molecular weight (g).

## 3.4 Instrumental

### 3.4.1Atomic Absoption Spectroscopy

Atomic Absoption is employed for the analyze of sodium content.

### 3.4.2 X-ray Diffraction Spectroscopy

X-ray (XRD) analysis is performed with SIEMENS XED D5000, accurately in the 5-60° 2θ. It is used to estimate the degree of crystallinity of zeolite.

### 3.4.3 Morphology

The shape and the distribution of size of crystal are observed by JEOL Scanning Electro microscope (SEM).