CHAPTER IV
ZEOLITE SYNTHESIS DIRECTLY FROM ALUMATRANE AND SILATRANE BY SOL-GEL PROCESS AND MICROWAVE TECHNIQUE

(Sensors and Actuators B. Chemical, Submitted)

4.1   Abstract

Alumatrane and silatrane synthesized by Oxide-One-Pot-synthesis (OOPS) process directly from silica and alumina are used to study the synthesis of zeolites., They were successfully used as precursors in the zeolite synthesis via the sol-gel process followed by microwave heating. Hydrolysis of the precursor mixture was studied by FTIR using alkaline hydroxide solutions as the hydrolytic agents. With NaOH as the hydrolytic agent, the transformation from amorphous metal oxide gel to crystalline aluminosilicate or zeolite was accomplished at the microwave temperature of 110°C, in agreement with the DSC transition at 106°C. The bigger the alkaline atom in alkaline hydroxide, the higher the microwave temperature needed. As a result, changing the hydrolytic agent to LiOH or KOH zeolite product was obtained at 90° or 150°C, respectively. The moisture absorption ability of synthesized Na A and K-H zeolites are higher than the commercial ones.

4.2   Introduction

Zeolites are used very widely in household products in chemical and petrochemical industry and even in space [1]. Depending on the framework structure and nature of zeolites or crystalline aluminosilicates, they are widely used in gas separation and refinery industries as catalysts, adsorbents and ion exchangers [2]. Most silica based zeolites synthesized using a template to form the desired pore structure using self assembly of inorganic-organic molecules. If organic-Inorganic self assembly is used then the inorganic parts need to be condensed at the head or the boundary of the organic potion to form the metal oxide linkage and grow further to produce crystalline product and form particle nuclei [3]. For self assembly to happen, a dilute solution of the precursor is needed. The relative rates of hydrolysis and condensation in the gel formation state is also of high importance in directing the framework structure. Moderate activity precursors provide time for a better formation meso-structure. The ideal precursor works well over a wide range of pH, especially from neutral to highly basic solutions. When dealing with more than one metal oxide, the relative and absolute reactivity of each precursor is extremely important since for the formation of a homogeneous product requires time for the proper insertion of each metal oxide into the framework.

In this work, our synthesized atranes, silatranes and alumatranes, were chosen as precursors for zeolite synthesis due to the common trialkoxyamine ligand that is able to coordinate with the metal atoms to form inorganic-organic micelles and to give moderately inert complexes in aqueous or basic solution [3-4]. Complex formation is thermodynamically stable in an aqueous-base system due to the possibility of expanding its coordination sphere. This phenomenon moderates the alkoxide reactivity towards the nucleophilic attack of water, as a result, it retards precipitation [3-5]. These properties make atranes attractive for forming meso-porous framework materials.

We have previously reported successful synthesis of ANA and GIS typed zeolites directly from alumatrane and silatrane [6]. The same method was adapted and applied to produce other types of zeolites by using different types of metal hydroxide as the hydrolytic agent in sol-gel state. In this work, the series of alkaline

metal hydroxides: LiOH, NaOH and KOH, were selected and used in producing LTA, Zeolite K-H and FAU type zeolites. Both LTA and zeolite K-H are widely used as molecular sieves, adsorbents, ion exchange and gas separation [7-11] while FAU type is used in catalytic cracking industry due to its ~7 Å pore size and high alumina content [12-14].

4.3   Experimental

4.3.1   Materials

Fumed silica (SiO_2, surface area 473.5 m^2/g, average particle size of 0.007 μm) and aluminum hydroxide hydrate ($Al(OH)_3$, surface area 50.77 m^2/g), were purchased from Sigma Chemical Co. and used as received. Triethanolamine (TEA, $N(CH_2CH_2OH)_3$), and triisopropanolamine (TIS, $N(CH_2CHCH_3OH)_3$) were supplied by Carlo Erba Reagenti and Fluka Chemical AG., respectively. Both were used as received. Ethylene glycol (EG, $HOCH_2CH_2OH$) was obtained from J.T. Baker Inc. and distilled using fractional distillation prior to use. Sodium hydroxide (NaOH) and sodium chloride (NaCl) were purchased from EKA Chemicals and AJAX Chemicals, respectively. Both were used as received. Acetonitrile (CH_3CN) was obtained from Lab-Scan Co., Ltd. and distilled using standard purification method.

4.3.2   Instrumentals

Fourier transform infrared (FTIR) spectroscopic analysis was conducted using a Bruker Instruments EQUINOX55 with a resolution of 4 cm^{-1}. The solid samples were prepared by mixing 1 wt % of sample with dried KBr, while the liquid samples were analyzed using Zn-Se window cell. Mass spectra were obtained using a VG Autospec model 7070E from Fison Instruments with the VG data system, using the positive fast atomic bombardment (FAB$^+$-MS) mode and glycerol as a matrix. CsI was used as a reference, while a cesium gun was used as an initiator. The mass range used was from m/e = 20 to 3,000. Thermal properties were analyzed using thermogravimetric analysis (TGA) and differential scanning calorimetry

(DSC) mode. TGA was performed with sample size of 10 – 20 mg using a Perkin Elmer instruments: TGA7 analyzer while DSC was conducted with sample size of 5–10 mg on Netzsch instrument: DSC200 Cell and TASC 414/3 controller at a heating rate of 10°C/min under nitrogen atmosphere. For liquid and gel samples, a high-pressure gold cell was used with the sample size of 10-20 mg. Simultaneous thermal analyzer (STA) was also employed for measuring the thermal stability and phase transformation of synthesized zeolite at a heating rate of 20°C/min under nitrogen atmosphere. The testing was carried out on Netzsch instrument:s STA409 EP, for which the sample size was in range of 10 - 50 mg using an alumina-crucible as the sample cell. Crystallinity of products was characterized with a Rigaku X-Ray Diffractometer at scanning speed of 5 degree/sec, using the Cu Kα, λ=1.54 Å emission. The working ranges were 5 – 90 and 5 – 50 theta/2 theta. SEM micrographs were obtained using a JEOL 5200-2AE scanning electron microscope. Electron Probe Microanalysis (EPMA) was used to analyze the sample at the micro-scale for both qualitative and quantitative elemental, using X-Ray mode detector (SEM/EDS; Energy dispersive spectroscopy). Particle size was determined using a Malvern instruments Mastersize X Ver.2.15,. Water was used as the mobile phase. Hydrothermal treatment by microwave heating technique' was conducted using MSP1000, CME Corporation (Spec. 1,000W and 2,450 MHz). Samples were heated in a Teflon-lined digestion vessel using inorganic digestion mode with time-to-temperature program. \

4.3.3 Precursor Synthesis

Silatrane was synthesized by mixing silicon dioxide, 0.10 mol, and triethanolamine, 0.125 mol, in a simple distillation set using 100 mL ethylene glycol, as solvent [15-16]. The two components were reacted for ten hours at the boiling point of ethylene glycol under nitrogen atmosphere to remove the byproduct water and ethylene glycol from the system. After stopping the reaction the rest of the ethylene glycol was removed under vacuum at 110°C. The product, a brownish white powder, was washed with dried acetonitrile for three times. Approximately 95% yield of white powder product was obtained and the product was characterized

using FTIR, TGA, DSC and FAB^{+}-MS. The synthesized product was named as SiTEA; FTIR: 3000-3700 cm^{-1} (w, intermolecular hydrogen bonding) 2860-2986 cm^{-1} (s, νC-H), 1244-1275 cm^{-1} (m, νC-N), 1170-1117 (bs, νSi-O), 1093 (s, νSi-O-C), 1073 (s, νC-O), 1049 (s, νSi-O), 1021 (s, νC-O), 915-940 (m, νSi-O-C), 785 and 729 (s, δSi-O-C) and 579 cm^{-1} (w, Si<---N). TGA: one mass loss transition at 390°C and 18.47 %ceramic yield corresponding to Si$((OCH_2CH_2)_3N)_2H_2$. DSC: 349°C (endothermic) and 380°C (exothermic). FAB^{+}-MS: approximately 3% of the highest m/e at 669 of Si$_3((OCH_2CH_2)_3N)_4H^{+}$ and 100% intensity at 323 of Si$((OCH_2CH_2)_3N)_2H_3{}^{+}$.

A similar process was used to synthesize alumatrane from aluminum hydroxide (0.1 mol) and triisopropanolamine (0.125 mol) [17]. The crude product was washed with dried acetonitrile for three times giving ~90% yield of product which was characterized using FTIR, TGA, DSC and FAB^{+}-MS. The synthesized product was named as AlTIS; FTIR: 3000-3700 cm^{-1} (s, intermolecular hydrogen bonding) 2860-2986 cm^{-1} (m, νC-H), 1649 (w, O-H overtone), 1244-1275 cm^{-1} (w, νC-N), 1130 (m, νC-O), 1102 (s, νAl-O-C), 1037 (m, νC-O), and 649 (s, δAl-O). TGA: two mass loss transitions at 139° and 393°C and 23.97 %ceramic yield corresponding to Al$(OCHCH_3CH_2)N$. DSC: 145° and 196°C (endothermic) and 380°C (exothermic). FAB^{+}-MS: approximately 4% of the highest m/e at 1292 of (Al$(OCHCH_3CH_2)_3N)_6H^{+}$ and 100% intensity at 216 of Al$(OCHCH_3CH_2)_3NH^{+}$.

4.3.4   Sol-Gel Process and Microwave Technique

SiTEA and AlTIS were mixed with NaOH solution at room temperature at a ratio of SiO$_2$:xAl$_2$O$_3$:yNa$_2$O:zH$_2$O (where $0 < x \leq 100$, $0 \leq y \leq 10$ and $63 \leq z \leq 1000$). The solution mixtures were aged for 12 h and then transferred to teflon containers for further hydrothermal treatment using microwave technique. The solution mixtures containing various ratios of SiO$_2$:xAl$_2$O$_3$:yNa$_2$O:zH$_2$O were hydrothermally treated for various times and the resulting white powder products were washed three time using distilled water. The products were finally dried overnight at 75°C. The gel started to form at SiO$_2$:Na$_2$O ratio of 1:0.0069, however, the best ratio of SiO$_2$:Na$_2$O for synthesizing zeolites was ranged from 1:1 to 1:3. To

obtain homogeneous dispersion of powder product in the solution mixture, water needed to be started at y = 63, and the suitable $SiO_2:H_2O$ ratio for synthesizing zeolites was 1:410. Since, there was no carbon component left in the pores or the framework of synthesized products, (verified by SEM/EDS and TGA) calcination of the products was not necessary.

4.3.5   Moisture Absorption

Crucibles were heated at 350°C for 10 h. and cooled to 120°C in a hot-air oven. They were then moved and kept in a dessicator until use. The samples and the dried crucibles were weighed and then heated at 350°C for 10 h. They were cooled to 120°C in oven and then kept in dessicator until they cooled to room temperature. The dried samples were weighed and dried weight of sample (W_1) was calculated. The dried samples were then placed in a closed humidification chamber for 15 h. to overnight at 40°C and then weighed to determine the mass of water adsorbed (W_2). The moisture absorption ability was calculated per unit mass of the sample (W_2/W_1).

4.4   Results and Discussion

4.4.1   Sol-Gel Process

In this work, MOH solution (where M = Li, Na, K) was used in the sol-gel process [15]. The framework structure of aluminosilicate is formed by joining tetracoordinated $(SiO_4)^{4-}$ with tetracoordinated $(AlO_4)^{5-}$ resulting in a myriad of lattice architectures which consist of channels and cages with different types of connectivity [18]. Negative charges on Al-atoms in the framework are stabilized by the Li, Na or K cation in the system.

4.4.2   Transformation to Aluminosilicate

After the gel was formed, the gel transformation to aluminosilicate by hydrothermal treatment was first studied using DSC high-pressure cell (Figure 1) to investigate where the maximum transformation should be.   Based on the gel

formation study it was found that the transformation was endothermic, in agreement with Yang [19] due to the dissolution of amorphous metal-oxide network and crystallization of aluminosilicate [20-21]. The transitions for all hydrolytic agents started at ~103°C and the maximum transformation occurred at ~106°C. Interestingly, the overall energy consumption increased with the size of the alkali-loading cation (steric effect).

As expected, transformation of the gel into crystalline products indeed depends on the metal hydroxide [22]. The higher the metal hydroxide concentration used, the higher the crystallinity. This is due to the negative charges in the framework structure that need to be stabilized by alkaline cations [23]. Moreover, the nucleation rate also depends on the type and metal hydroxide concentration. Though, the gel transformation temperature of each hydrolytic agent obtained from DSC occurred in the same temperature range, different energy consumption of each hydrolytic agent practically caused the formation of crystalline aluminosilicate products at different temperatures. Coincidentally, the temperature of zeolite product formation also increased with the cation size. K-zeolite was obtained at the highest temperature, 150 °C, while Na-zeolite and Li-zeolite were produced at 110° and 90°C, respectively. However, a suitable temperature for making perfect Li-zeolite crystals was found to be 105°-110°C.

Na A or LTA typed zeolite, having cell structure and crystal morphology unit, as illustrated in Figure 2, was obtained at the loading ratio of $SiO_2:Al_2O_3:3Na_2O:410H_2O$, after heating for 180 min at 110 °C. The reaction started from almost a clear solution and the white precipitated product was formed after the hydrothermal treatment, the gel formation and transformation to aluminosilicate thus occurred during hydrothermal treatment [24-25]. The Si:Al:Na ratio of the synthesized product obtained from EDS-SEM was at ~1:1:1.25 and its XRD matched the spectrum of Si:Al XRD, PDF#39-0222, which had ratios of Si:Al:Na = 1:1:1. The higher Na/Si ratio must come from high NaOH concentration used. The OH⁻ anions in solution might interact with zeolite framework to form the siloxy groups ($\equiv SiO^-$) the charges of which must be balanced by the extra Na^+ cations [26]. The synthesized product did not have any carbon component left,

(confirmed by EDS-SEM and STA (no carbon or carbon oxidation peak observed)). STA also indicated that the thermal stability of the synthesized product was around 600 °C, at which temperature a slow phase transformation from LTA to Sodium Aluminum Silicate (PDF#11-0220, $NaSiAlO_4$) occurred, Figure 3, due to the collapse of LTA.

Using KOH as the hydrolytic agent, zeolite K-H was produced at the loading ratio of $SiO_2:0.1Al_2O_3:3K_2O:410H_2O$ and heating for 240 minutes at 150 °C. Due to a better atrane-K complex formation causing good dissolution of atrane [13], the reaction solution started from almost clear solution and then turned to white precipitated product after hydrothermal treatment. Matched XRD data indicated that the synthesized product was of the unknown structure type (PDF#16-0692, Si:Al:K = 2:1:1). The crystal morphology was needle-like. The needles appeared to aggregate into assemblies similar to flower petals, we named this zeolite PPC-ZM-1 (standing for "the first discovery new zeolite at the Petroleum and Petrochemical College") as shown in Figure 4. Si:Al:K ratio of the synthesized product, obtained from EDS-SEM was 1.98:1:0.82. In general, the cations are not the part of framework and instead sit near the oxygen rings at the corner of ring or the window of unitcell [27]. All of the aluminum and silicon atoms are buried in tetrahedra of oxygen atoms and are not exposed to the surface hence the surface is composed of oxygen atoms and the cations. Owing to the larger size of K^+, some places of two adjacent Al-positions might occupy by one K^+ leaving one extra free aluminum anion. Thus, this zeolite product adsorbed water faster than Na A zeolite, as seen in Table 1. The thermal stability of the synthesized product was about 600 °C, above which temperature the phase transformation to amorphous aluminosilicate occurred very slowly (Figure 5).

Hydrolytic agent LiOH started to form a crystalline product at 90 °C and loading ratio of $SiO_2:0.5Al_2O_3:3Li_2O:410H_2O$. However, to synthesize FAU typed zeolite using Li_2O/SiO_2 ratio equal to one the mixture must be heated at 110°C for 120 min. Matched XRD data indicated the mixture of FAU (PDF#: Si:Al:Li = 2:1:1) and LZ-200 (PDF#: Si:Al = 1:1:1.06), while the EDS-SEM result gave the Si:Al ratio at ~1.7:1. The morphology of the synthesized product powder was disk-

like mixed with a very small portion of round balls which might be LZ-200 (Figure 6). The synthesized product was thermally stable up to ~680 °C above which it changed to lithium aluminate silicate (PSF#26-0839: $LiAlSiO_4$), as illustrated in Figure 7.

Agglomerated particle size and particle size distributions of both Na A and K-H zeolites have similar profile, but different from FAU (Figure 8). Na A or K-H have bimodal particle size distributions, ~11.3 % having diameters less than 2.90 μm and an average agglomerated particle size of ~8.21 μm (one single particle size obtained from SEM was at ~5 μm (Figure 2)). For zeolite K-H, 14.8% was less than 3.27 μm and the maximum population was at ~10.84 μm which is almost the same size as observed from SEM (Figure 4). FAU also has a bimodal size distribution, but the distribution profile was totally different.

4.4.3 Moisture Absorption Testing

Comparatively, the absorption ability of Zeolite K-H was higher than the others while that of LTA and FAU were almost the same. Zeolite K-H can adsorb ~25% of sample weight while LTA and FAU can adsorb ~16%, see table 1.

4.5 Conclusions

Silatrane and alumatrane can go through the sol-gel process followed by hydrothermal treatment in the presence of metal hydroxide to provide zeolites narrow particle size distributions, as observed by SEM and particle size analyzer. The transformation seen by the DSC occurred at the same temperature range for all hydrolytic agents, but the energy requirement for the KOH system was higher than that of NaOH and LiOH systems. The energy required to form zeolites is in the following order; K-H > Na A > FAU. FAU has the smallest particle size while the moisture adsorption ability of zeolite K-H is the highest. Different types of hydrolytic agents provided different type of products.

4.6 Acknowledgement

4.7 References

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4.8   Biography

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Table 4.1   Moisture adsorption of synthesized zeolites

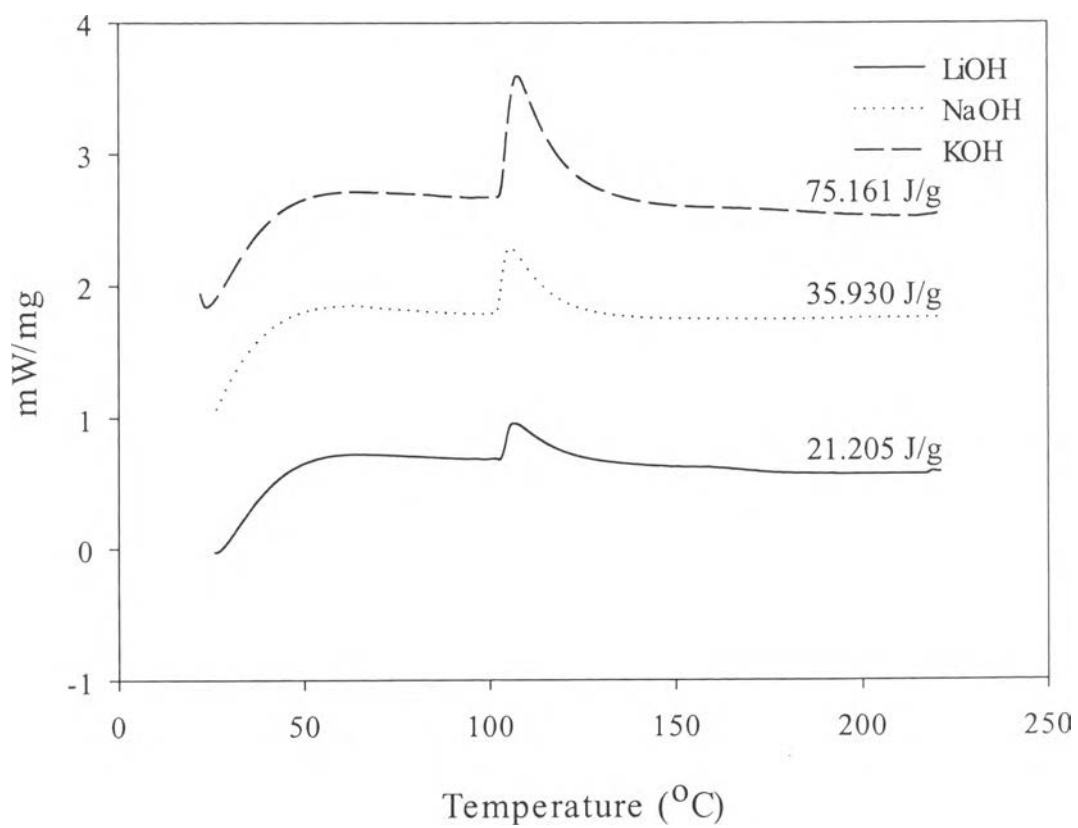| Synthesized Zeolite	LTA	Zeolite K-H	FAU
Moisture Absorption (gH_2O/g)	0.1598	0.2463	0.1574
Ratio	1.02	1.56	1.00

Figure 4.1 Thermal property of gel transformation to aluminosislicate using high pressure DSC cell at heating rate of 10°C/min and $1SiO_2:0.5Al_2O_3:0.7Na_2O: 410H_2O$
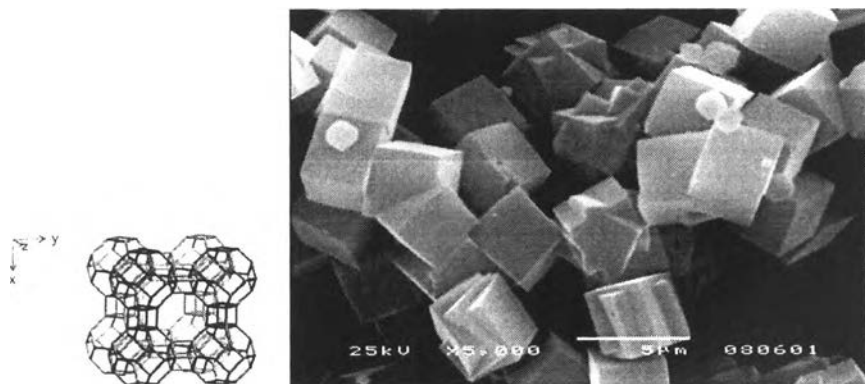
Figure 4.2 Unit cell structure and crystal morphology of Na A zeolite synthesized from $SiO_2:Al_2O_3:3Na_2O:410H_2O$ and 110°C for 180 min in $NaOH/H_2O$ system
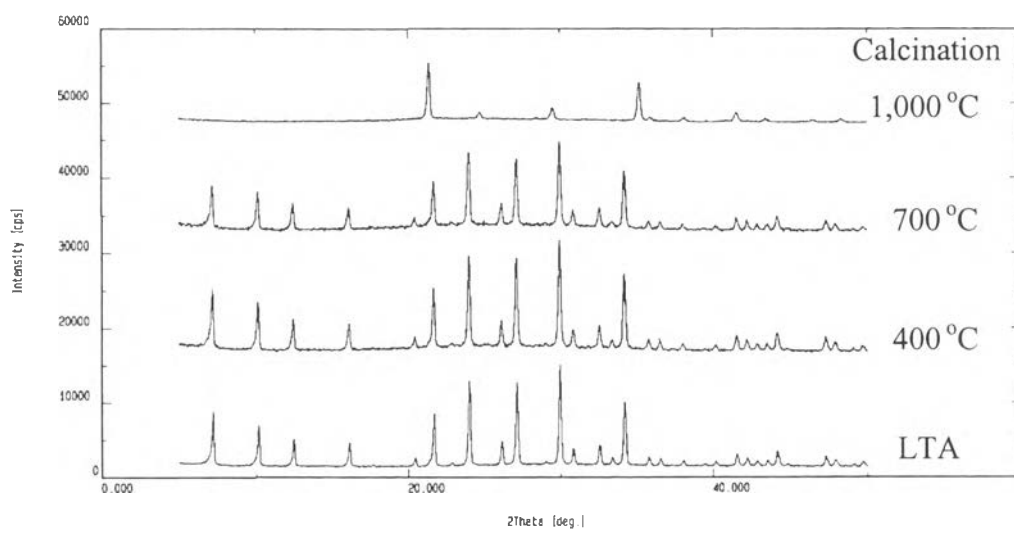
Figure 4.3   XRD spectra of calcinated Na A zeolite at various temperatures (room temperature to 1000 °C)
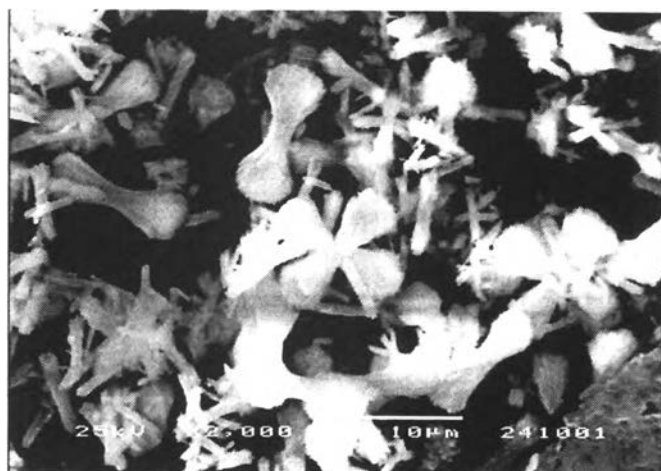
Figure 4.4   Unit cell structure and crystal morphology of Zeolite K-H synthesized from $SiO_2:0.1Al_2O_3:3K_2O:410H_2O$ and 150°C for 240 min in NaOH/H$_2$O system
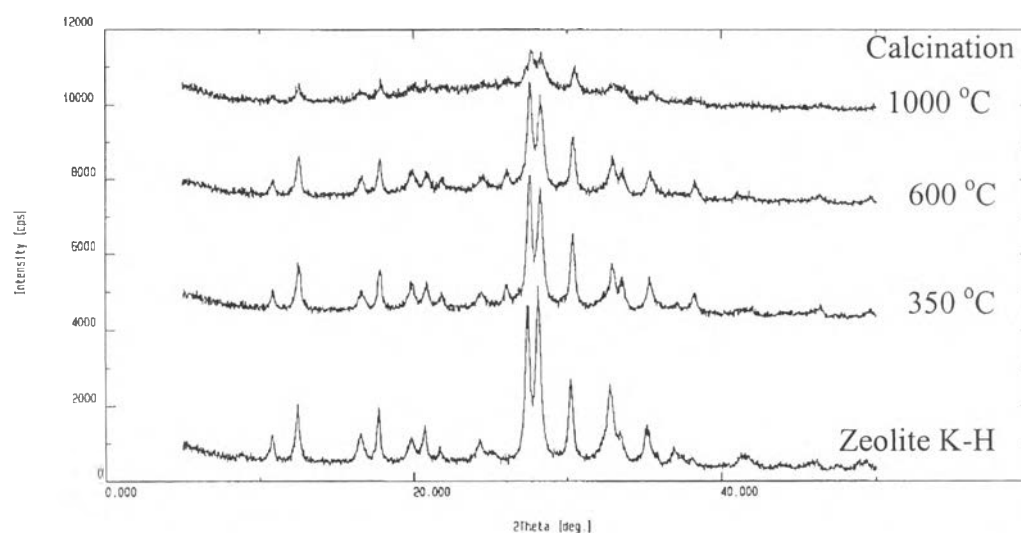
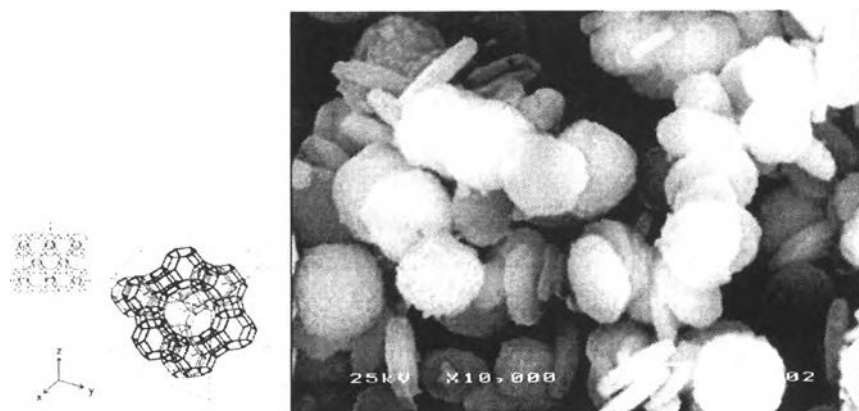**Figure 4.5** XRD spectra of calcinated Zeolite K-H at various temperature (room temperature to 1000 °C)

Figure 4.6 Unit cell structure and crystal morphology of FAU type zeolite synthesized from $SiO_2:0.5Al_2O_3:1Li_2O:410H_2O$ and 110°C for 120 min in NaOH/H_2O system
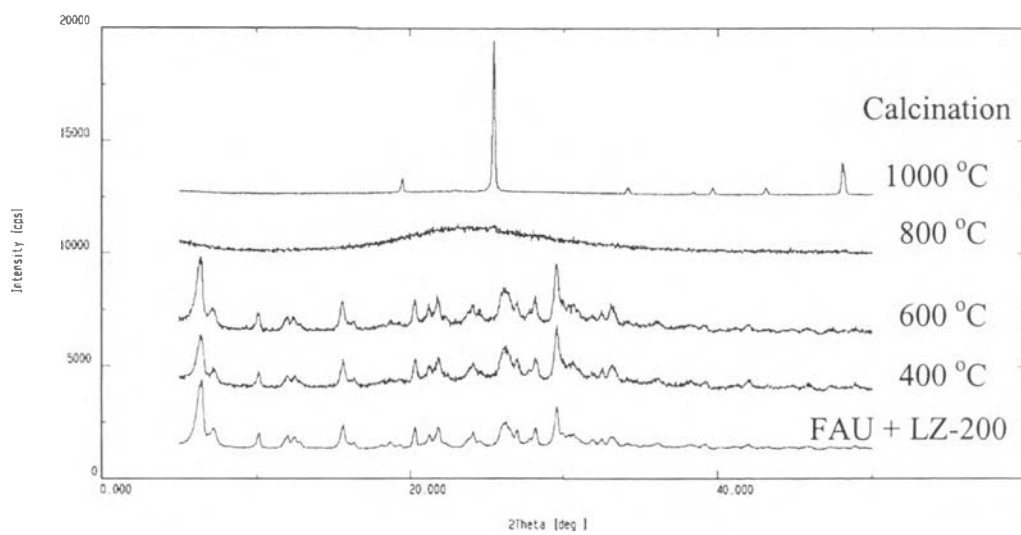
Figure 4.7 XRD spectra of calcinated FAU type zeolite at various temperature
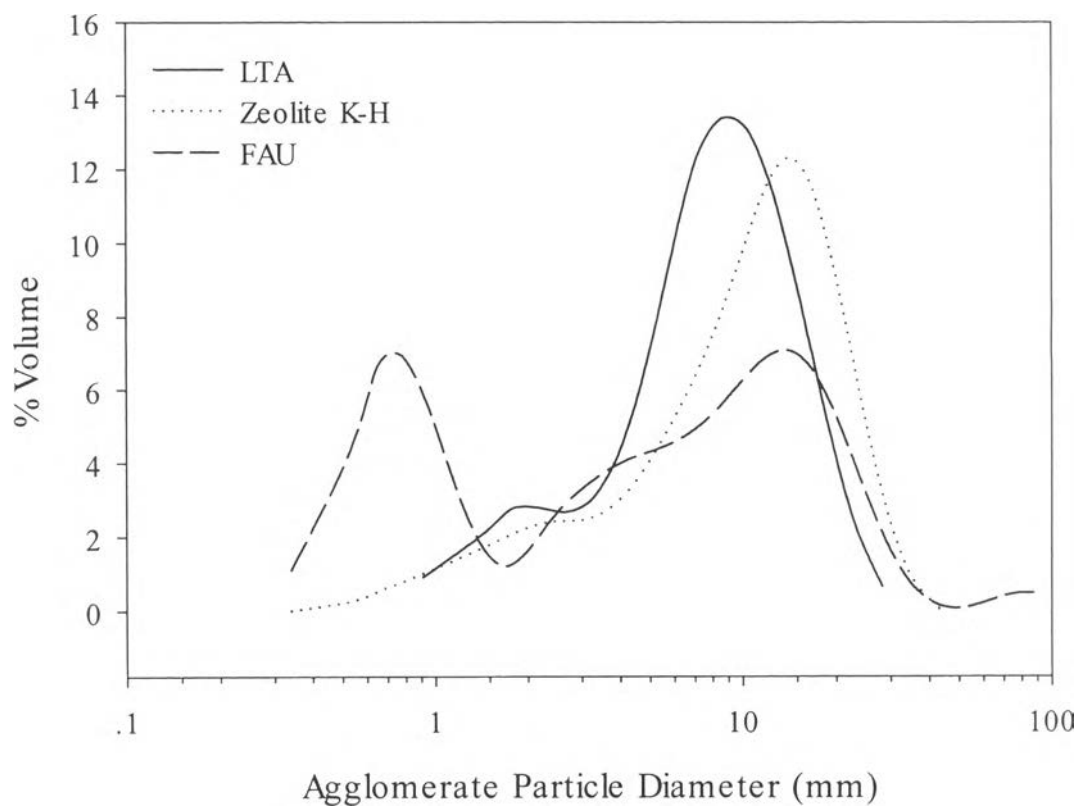
(room temperature to 1000 °C)

Figure 4.8   Agglomerated particle size distribution of synthesized zeolites