CHAPTER III
EXPERIMENTAL

3.1 Materials

Fumed silica (SiO_2, 99.8%, Nippon Aerosil, Japan), aluminium hydroxide ($Al(OH)_3$, Sigma, USA), lithium hydroxide (LiOH, >99.5%, Fisher Chemicals, UK), sodium hydroxide (NaOH, 99%, RCI Labscan, Thiland), potassium hydroxide (KOH, Sigma-Aldrich, USA), hydrogen gas (H_2), carbon monoxide gas (CO), oxygen gas (O_2), helium gas (He), nitrogen gas (N_2), gold(III) chloride trihydrate ($HAuCl_4 \cdot 3H_2O$, Sigma-Aldrich, USA), hexachloroplatinic(IV) acid hexahydrate ($H_2PtCl_6 \cdot 6H_2O$, Merck, Germany) were used with no further purification.

3.2 A-Type Zeolite Synthesize

3.2.1 Synthesis of Li-A Zeolite

To synthesize Li-A zeolite, SiO_2 and $Al(OH)_3$ were mixed with LiOH at the molar ratio of SiO_2:0.5Al_2O_3:3Li_2O:410H_2O (Sathupunya *et al.*, 2004). The mixture was aged for 12 h at room temperature to obtain fully gel formation and brought to hydrothermal treatment using microwave technique for 10 h. The synthesized product was washed using distilled water three times and dried at 60 °C overnight. The obtained white powder product was characterized using SEM and XRD.

3.2.2 Synthesis of Na-A Zeolite

To synthesize Na-A zeolite, SiO_2 and $Al(OH)_3$ were mixed with NaOH at the molar ratio of SiO_2:Al_2O_3:3Na_2O:410H_2O (Sathupunya *et al.*, 2003). The mixture was aged for 12 h at room temperature to obtain fully gel formation and brought to hydrothermal treatment using microwave technique for 10 h. The synthesized product was washed using distilled water three times and dried at 60 °C overnight. The obtained white powder product was characterized using SEM and XRD.

3.2.3 Synthesis of K-A Zeolite

To synthesize K-A zeolite, SiO_2 and $Al(OH)_3$ were mixed with KOH at the molar ratio of SiO_2:0.1Al_2O_3:3K_2O:410H_2O (Sathupunya *et al.*, 2004). The mixture wass aged for 12 h at room temperature to obtain fully gel formation and brought to hydrothermal treatment using microwave technique for 10 h. The synthesized product was washed using distilled water three times and dried at 60 °C overnight. The obtained white powder product was characterized using SEM and XRD.

3.3 Catalyst Preparation

All catalysts were prepared by the impregnation method, using A-type zeolites as supports (Luengnaruemitchai *et al.*, 2008). Aqueous solution of $HAuCl_4 \cdot 3H_2O$, containing 1wt% gold, and $H_2PtCl_6 \cdot 6H_2O$, containing 1wt% platinum, were impregnated onto the synthesized zeolite supports. All catalysts were dried in the air at 110 °C overnight and calcined at 500 °C for 5 h. Prior to each catalyst measurement, the catalyst was grounded and sieved to an 80–120 mesh size. The loaded gold and platinum contents were determined by AAS.

3.4 Catalyst Characterization

The crystallinity of zeolite supports and metal loaded supports were analyzed using Rigaku X-ray diffraction (XRD) system quipped with a RINT- 2200 wide angle goniometer using CuK_α radiation (1.5406 A°) and a generator voltage and current of 40 kV and 30 mA, respectively. Hitachi, TM3000 scanning electron microscope (SEM) provides the support morphology. The metal content was determined by Varians, Spectra AA 300 atomic adsorption spectrophotometer (AAS). Transmission electron microscopy (TEM) images were taken with JEOL, JEM-2010.

3.5 Activity Measurement

All catalytic activity measurement for CO oxidation was performed in a fix-bed U-tube micro-reactor with 100 mg of the catalyst (Luengnaruemitchai *et al.*, 2008). The catalyst activity was investigated in a temperature range of 100°–350°C. The inlet gas contained 1% CO, 1% O_2, balance in He, with a total rate of 50 mL/min under atmospheric pressure. The outlet gasses from the reactor were analyzed by an on-line GC equipped with a packed carbosphere and TPD detector.