



CHAPTER III EXPERIMENTAL

3.1 Equipments and Materials

Equipments:

3.1.1 Fourier Transform Infrared Spectrophotometer (FT-IR)

Infrared spectroscopy provided functional groups information on the vibration of the precursors (silatrane, and molybdenum glycolate) which the spectra were obtained using a Nicolet, NEXUS 670 with a resolution of 4 cm^{-1} . The solid samples were mixed and pelletized with dried KBr.

3.1.2 Thermal Gravimetric Analysis (TGA)

TGA thermograms were carried out using TG-DTA (Pyris Diamond Perkin Elmer) with a heating rate of $10^\circ\text{C}/\text{min}$ in the range of room temperature to 750°C under nitrogen atmosphere to determine the thermal stability of silatrane, and molybdenum glycolate.

3.1.3 X-Ray Diffraction (XRD)

X-ray patterns were carried out to characterize crystallinity on Rigaku X-ray diffractometer with $\text{CuK}\alpha$ as a X-ray source. Sample was spread on a glass slide specimen holder. A 2θ range from 1.8° to 8° was investigated using a rate of $0.02^\circ/\text{min}$.

3.1.4 Diffuse Reflectance UV-VIS Spectroscopy (DRUV)

DRUV was extensively used to detect the framework and extra-framework molybdenum species using SHIMADZU UV 2550-VISIBLE spectrophotometer in the range of 200 to 600 nm.

3.1.5 Surface Area and Average Pore size

Surface area and average pore size were measured by BET method using a Quantachrome JR. (Autosorb 1). The catalyst was degassed at 250°C for 10 h before analysis.

3.1.6 Scanning Electron Microscope (SEM)

Morphology of the synthesized materials were observed by secondary electron imaging with a JEO 5200-2AE scanning electron microscope (SEM).

3.1.7 Gas Chromatography (GC)

Products of the epoxidation reaction were analyzed by using a gas chromatography, Agilent 6890N, with a capillary column (ZB-WAX, 30 M × 0.25 mm) and FID detector.

Chemicals:

Fumed silica (SiO₂, 99.8%) (Sigma-Aldrich), triethanolamine (TEA) (Carlo Erba), ethylene glycol (J.T.Baker, USA), acetonitrile (Labscan, Asia), sodium sulfate (Ajax Finechem), molybdenum(VI) oxide (Fluka), hexadecyltrimethylammonium bromide (C₁₆TMAB) (Sigma-Aldrich), sulfuric acid (Labscan, Asia), sodium hydroxide (Labscan, Asia), hydrogen peroxide (Labscan, Asia), and styrene monomer (Labscan, Asia) were used as received.

3.2 Methodology

3.2.1 Preparation of silatrane precursor (Charoenpinijkarn *et al.*, 2001).

Silatrane was synthesized via the Oxide One Pot Synthesis (OOPS) process by mixing SiO₂ (6.0 g) and TEA (18.6 g) in a simple distillation set using EG as solvent. Reaction was taken place at the boiling point of EG under N₂ atmosphere and kept running for 10 hours. Removal of EG under vacuum was the next step to obtain crude solid product, followed by washing with acetonitrile to obtain pure silatrane.

3.2.2 Synthesis of mesoporous SBA-1 (Tunglumlert *et al.*, 2007)

Solution A was prepared by adding C₁₆TMAB to 30 ml of water and stirred for 30 min to obtain a clear solution. Solution B was prepared by adding 5 mmol silatrane and 1.7 mmol NaOH to 14 ml of 0.3 M H₂SO₄, and stirred for 30 min to obtain a homogeneous solution. The solution B was then added to the solution A under vigorous stirring and continuously stirred for 4 h. The mixture was then centrifuged to get rid of the unreacted materials. The obtained solution was aged at

room temperature for 3 days to form precipitate. The product was filtered and washed with distilled water. Pure and dried product was finally calcined at 560°C using a Carbolite Furnace (CFS 1200) with a heating rate of 0.5°C/min to remove template.

3.2.3 Synthesis of molybdenum glycolate (Sutara *et al.*, 2004)

Molybdenum glycolate was synthesized by mixing MoO₃ (4.29 g) and EG (50 ml) with vigorous stirring and heating at the boiling point of EG under N₂ atmosphere for 15 min. The obtained solution was centrifuged to separate the unreacted MoO₃. The solution part is left at room temperature for 3 days to obtain molybdenum glycolate solid, followed by washing with acetonitrile and drying in a dessicator.

3.2.4 Synthesis of Mo-SBA-1

Similar to the synthesis of SBA-1, both solutions A and B were prepared, and after adding solution B into solution A, molybdenum glycolate precursor was added into the mixture. The mixture was stirred for 4 h, followed by centrifuging to get rid of the unreacted materials. The obtained solution was aged for 3 days to obtain crude product. This crude product was filtered and washed with distilled water to obtain white solid. The white solid was calcined at 560°C to result in Mo-SBA-1. Mo-SBA-1 was also prepared by wetness impregnation method by using molybdenum glycolate as the molybdenum source, followed by oven drying at 100°C and calcination at 560°C.

3.2.5 Epoxidation Reaction of Styrene (Wang *et al.*, 2002)

The epoxidation reaction of styrene was carried out by using a batch-type reactor. Catalyst (x g), styrene monomer (10 mmol), an H₂O₂ oxidant (9.8 mmol, 30 wt% aqueous solution) and the solvent acetonitrile (10 ml) were added into a glass flask. The factors studied were amount of catalyst used, reaction time, reaction temperature, and amount of Mo loaded. After the catalyst was filtered, the reaction mixture was analyzed by GC to observe the reactants and the products. The conversion of the styrene was calculated on the basis of the amount of styrene used.