

## CHAPTER III

### EXPERIMENTAL

#### 3.1 Materials

Fumed silica ( $\text{SiO}_2$ ) from Aldrich Chemical Co., UHP grade nitrogen ( $\text{N}_2$ , 99.99% purity) from Thai Industrial Gases Public Company Limited (TIG), ethylene glycol (EG), triethanolamine (TEA) and acetonitrile ( $\text{CH}_3\text{CN}$ ) from Labscan were used for silatrane synthesis. Ferric Chloride ( $\text{FeCl}_3$ ) from Aldrich Chemical Co., hexadecyltrimethyl ammonium bromide (CTAB) from Sigma Chemical Co. and sodium hydroxide (NaOH) from Univar were used for Fe-MCM-41 synthesis. Styrene monomer and dimethylformamide (DMF) from Labscan, hydrogen peroxide from Carlo Erba were used for activity study.

#### 3.2 Synthesis

Following the method of Wongkasemjit, silatrane, as silicon source, was synthesized directly from inexpensive and widely available starting materials,  $\text{SiO}_2$  and TEA, via the Oxide One Pot Synthesis (OOPS) process [12]. Fe-MCM-41 was synthesized via the one-step synthesis using silatrane and  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  as silicon and iron sources, respectively. The composition of the reactants is followed the previous study on the synthesis of extremely high surface area MCM-41 [13]. The formula used was  $x\text{silatrane} : y\text{FeCl}_3 \cdot 6\text{H}_2\text{O} : 0.014\text{TEA} : 0.5\text{CTAB} : 0.001\text{NaOH} : 0.36\text{H}_2\text{O}$ . To study the optimal condition for Fe-MCM-41 synthesis, iron concentration, synthesis time, reaction temperature, calcinations rate as well as metal amount were varied.

#### 3.3 Characterization

FTIR spectroscopic analysis was conducted on a Bruker Instrument (EQUINOX55) with a scanning resolution of  $4 \text{ cm}^{-1}$ . Thermal properties were analyzed by thermogravimetric analysis (TGA) on Du Pont Instrument TGA 2950 with the  $\text{N}_2$  flow rate of 50 ml per min. Mesoporous product was characterized using

a Rigaku X-ray diffractometer at a scanning speed of 2°/sec and CuK $\alpha$  as source. Diffuse reflectance UV-vis (DR-UV) spectra were recorded on a SHIMADZU UV-2550 UV-VISIBLE spectrophotometer. Powdery sample was loaded in a teflon cell, and the spectra were collected in 190-600 nm range with a reference of BaSO<sub>4</sub>. Surface area and average pore size were measured by BET method using a Quantasorb JR. (Autosorb-1). The product was degassed at 250°C for 10 hrs before analysis. ESR spectra were measured at X-band, ~9 GHz, on a ESPRIT-425 vol.604 spectrometer. The sample was placed in a quartz tube with an inner 3 mm in diameter and measured at ambient temperature. X-ray fluorescence spectroscopy or XRF (Bruker Model SRS 3004) was used to determine the amount of Fe waiting for the specification.

### 3.4 Catalytic Reaction

The epoxidation of alkene (styrene) was carried out using a batch-type reactor. Catalyst (x g.), styrene (10 mmol), H<sub>2</sub>O<sub>2</sub> (9.8 mmol, 30 wt% aqueous solution) and DMF (10 mL) were added in a glass flask. The reactant mixture was stirred vigorously. The factors studied were amount of catalyst, temperature, time, and amount of iron loaded. Generally, styrene oxide and benzaldehyde were two main products in the epoxidation of styrene with H<sub>2</sub>O<sub>2</sub> over Fe-MCM-41 [8]. However, styrene glycol and benzoic acid were also formed with low selectivity [8]. All liquid products were quantified using a gas chromatograph with a capillary column (DB-WAX, 30 M $\times$ 0.25 mm) and a FID detector. The consumption of H<sub>2</sub>O<sub>2</sub> was determined by iodometric titration.