CHAPTER III
EXPERIMENTAL

3.1 Materials

- Fume silica (99.8% SiO_2) : Sigma-Aldrich
- Cetryltrimethylammonium bromide (CTAB) : Sigma-Aldrich
- Sodium hydroxide (NaOH) : Sigma-Aldrich
- Ceriumnitrate hexahydrate (99%) : Sigma-Aldrich
- Ethylene glycol (EG) : J.T.Baker
- Triethanolamine (TEA) : QREC
- Acetronitrile : Labscan
- Ethanol : Merck
- Deionized water

3.2 Equipments

3.2.1 Field emission scanning electron microscope (FE-SEM) / Hitachi FE-SEM S4800

Field-emission scanning electron microscopy was used to determine the size, morphology and also the pore system of particles.

3.2.2 Transmission electron microscope (TEM) / JEOLJEM-2100

TEM images was used to provide further exploration in morphology and structure, including dimension of samples.

3.2.3 Thermogravimetric analyzer (TGA)

Thermogravimetry was used to analysis thermal properties by measuring the change in mass of a solid material as a function of temperature or time.

3.2.4 X-ray diffractometer (XRD) / Rigaku DMAX 2200 HV

X-ray diffraction was used to identify the crystalline phases present in the structure. The diffraction pattern is the fingerprint of any crystalline phase.

3.2.5 N_2 adsorption/desorption / Quantachrome Autosorb-1

Sorption measurement was used to determine into the pore structure of porous material such as the inner pore surface area, the pore volume and the pore diameter distribution.

3.2.6 X-ray fluorescence spectrophotometer (XRF) / AXIOS PW 4400

The X-ray fluorescence spectrophotometer (XRF) was employed to observe the element contents in samples.

3.2.7 Temperature programmed reduction (TPR)

Temperature programmed reduction was used to analyze the reducibility of metal oxides, mixed metal oxides and metal oxides dispersed on a support.

3.2.8 Fourier transforms infrared spectrophotometer (FT-IR)

Fourier transform infrared spectrophotometer is used to investigate the functional groups of chemical composition.

3.3 Methodology

3.3.1 Synthesis of silatrane

The synthetic method was followed Wongkasemjit's method by mixing 0.1 mol silica, 0.125 mol triethanolamine, and 100 ml ethylene glycol. The mixture was refluxed at 200 °C under nitrogen atmosphere for 10 hours in oil bath before excess ethylene glycol was removed at 110 °C under vacuum. The white silatrane product was purified by acetronitrile and vacuum-dried overnight. The obtained product was characterized by FT-IR at a resolution of 2 cm^{-1} to investigate functional group and also analyzed % ceramic yield from mass loss by TGA using a heating rate of 10 °C/min from room temperature to 650 °C in nitrogen atmosphere.

3.3.2 Synthesis of mesoporous MCM-48

Following Wongkasemjit's method, CTAB, used as surfactant, was dissolved in water and 2M NaOH with heating at 50 °C to dissolve. Then, the

silatrane was added to the solution and stirred for 1 h. The molar composition of the mixture was $1.0SiO_2$:0.3CTAB:0.5NaOH:$62H_2O$. The mixture was treated at 130°–150 °C for 16 h by using a Teflon-lined stainless steel autoclave to obtain solid product. The product was collected by filtration and dried overnight at ambient conditions. The surfactant was removed by calcinations at 550 °C for 6 h at a heating rate of 0.5 °C/min to obtain MCM-48. The obtained product, MCM-48, was characterized by FE-SEM and XRD carried out in a range of 2θ = 2–6° at a scanning speed of 1 °C/min.

3.3.3 Synthesis of ordered mesoporous ceria

The MCM-48 used as silica hard template and inorganic cerium nitrate (50, 60, 70, and 80% weight of ceria) were dissolved in 5 ml of ethanol. After stirring (30 min, 1, 2, and 4h), the ethanol in the mixture was removed by evaporation in an oven (room temperature, 50°, 100 °C). The process was repeated to get the 2 and 3 filling cycles of ceria. The obtained powder was heated in a ceramic crucible at 550 °C for 6 h to decompose the nitrate species. The silica hard template was removed by using 2M NaOH at 50 °C for 3 times and the mixture was centrifuged to obtain the product. The product was washed by deionized water and centrifuged until the washing was neutral and dried at 100 °C. The obtained products were characterized by XRD, FE-SEM, TEM, XRF and N_2 adsorption/desorption.

3.4 Temperature Programmed Reduction

Temperature programmed reduction with hydrogen was performed in a flow reaction system using 5% hydrogen in argon used as a carrier gas (flow rate 10ml/min). The ordered mesoporous ceria, 0.05 g, was heated from room temperature to 900 °C with a linear ramp rate of 10 °C/min.