CHAPTER III

EXPERIMENTAL

3.1 Materials

Molybdenum (VI) oxide (MoO_3) was purchased from Aldrich Chemical Co. Inc. (USA) and used as received. Ethylene glycol (EG) and acetonitrile were purchased from Farmitalia Carlo Erba (Barcelona) and Lab-Scan Company Co.Ltd., respectively. Both were distilled using standard methods before use.

3.2 Instruments

3.2.1 Fourier Transform Infrared Spectroscopy (FTIR)

Infrared absorption spectra (IR) were recorded on a Nicolet spectrometer, Nexus 670 model, with spectral resolution of 4 cm^{-1} using KBr mixed with sample.

3.2.2 X-Ray Diffraction (XRD)

Characterization of crystal structure of products were obtained from a Rigagu X-ray diffractometer (XRD) system equipped with a RINT 2000 wide angle goniometer and a Cu tube for generating a CuK α 1 radiation (λ = 1.54 A^{o}) was used to obtain the X-ray diffraction patterns at a generator voltage of 40 kV and a generator current of 30 mA. Nickel filter was used as the K_{β} filter. The goniometer parameters were divergence slit = 1° (2θ); scattering slit = 1° (2θ); and receiving slit = 0.3 nm. Sample was spread on a glass slide. A scan speed of 5° (2θ)/min with a scan step of 0.02° (2θ) was used during a continuous run in the 5° to 80° (2θ) range.

3.2.3 Scanning Electron Microscope

The scanning electron micrographs were carried out to identify the

microstructure of a sample. The samples were characterized using a JEOL 5200-2AE scanning electron microscope (SEM).

3.2.4 Thermo gravimetric Analysis (TGA)

Thermo gravimetric analysis was carried out on a Perkin Elmer TG-DTA pyres diamond over 30°–800°C at a heating rate of 10°C/min under nitrogen atmosphere.

3.2.5 Surface Area Measurement

The surface area of all samples was measured by the five- point BET method using a Quantachrome Corporation Autosorp I. Before the measurement, a sample was outgassed by heating at 200 ^{0}C for 6 h under vacuum to eliminate volatile adsorbents on the surface.

3.3 Methodology

3.3.1 Synthesis of Molybdenum Glycolate

Following the method of Wongkasemjit [15], molybdenum glycolate was synthesized directly from inexpensive and widely available staring materials, MoO_3 and ethylene glycol, via the oxide One Pot Synthesis (OOPS) process. The white product was characterized using FTIR and TGA.

3.3.2 Sol-gel Process of Molybdenum Glycolate

Molybdenum glycolate was mixed with either 2 M hydrochloric acid solution, 2 M nitric acid or non-acid solution. Various volume ratios of H_2O/HNO_3, H_2O/HNO3/MeOH, hydrolysis ratios, calcinations temperatures, times and heating rates were investigated. The mixture was vigorously stirred to form sol, followed by heating the sol at 40°, 50° and 60°C for 12, 2 and 1 h to form gel. The gels were dried at 110°C for 12 h before calcinations at temperature varied from 300° to 600°C for calcinations time ranging from 1 to 7 h and heating rate ranging from 0.25 to 1.0°C/min. The calcined product, molybdenum oxide, was characterized using XRD, SEM and BET.