CHAPTER III
EXPERIMENTAL

3.1 Materials

The starting material tin oxide (SnO_2) with a surface area of 9.8 m^2/g was purchased from Sigma-Aldrich Laborchemikalien GmbH. Ethylene glycol (EG) was obtained from Malinckrodt Baker, Inc. (USA). Triethylenetetramine (TETA) was purchased from Facai Polytech. Co. Ltd. (Bangkok, Thailand). Acetronitrile was supplied from Lab-Scan Company Co. Ltd. All chemicals were used as received.

3.2 Instruments

3.2.1 Fourier Transform Infrared Spectroscopy (FTIR)

Infrared absorption spectra (IR) were recorded on a Nicolet spectrometer 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 70° (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°–900°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 seven- point BET method using a Quantachrome Corporation Autosorp I. Before the measurement, a sample was outgassed by heating at 523 K for 4 h under vacuum to eliminate volatile adsorbents on the surface.

3.3 Methodology

3.3.1 Synthesis of Tin Glycolate via Oxide One Pot Synthesis (OOPS) process

Most importantly, tin glycolate is the newly synthesized and moisture stable precursor for the sol-gel process to obtain high surface area tin oxide. Tin glycolate was synthesized directly via the OOPS method from SnO_2, EG and TETA acting as base catalyst following the method for titanium glycolate synthesis. The mixture of SnO_2 (15.069g,0.1 mol) and TETA (14.62 g, 0.1 mol) was stirred vigorously in excess EG (100 ml) and heated up to 200°C using silicone oil bath under nitrogen atmosphere for 24 h. The mixture solution was centrifuged to separate unreacted SnO_2 and the solution was vacuum distilled to remove excess EG and TETA and giving product to precipitate off. The white solid product was washed with acetonitrile and dried in a vacuum desiccator. The white solid product was characterized using FT-IR, TGA, XRD and ^{13}C-NMR.

3.3.2 Sol-gel Processing of Tin Glycolate

Tin glycolate precursor 0.2217 g (0.0928 mmol) was dissolved in various HNO_3/H_2O ratio. The ratio of HNO_3/H_2O were varied by x μL of 8.0 M HNO_3 : yμL of water, where x:y = 1014:2535, 1014:2028 and 1014:1352. After we find optimum condition that tin glycolate in suitable solution to form gel at room temperature and dried at 110°C for 10 h, The dried powder was calcined at various temperature and time to study the crystallinity, morphology and surface area. The temperature used was varied from 300°C -900 °C and time from 2 to 6 h. After tin oxide powder was prepared via sol-gel process, it was characterized using SEM, TGA and XRD. The surface area was also measured using BET method.