



CHAPTER III METHODOLOGY

3.1 Materials and Instruments

3.1.1 Materials

2,5-dimethoxy aniline (Sigma Aldrich, AR grade, 98%) was used as a monomer. Oxalic acid (Ajax Finechem, AR grade), hydrochloric acid (Lab Scan, AR grade, 37%), sulfuric acid (Lab Scan, AR grade, 98%), nitric acid (Lab Scan), and acetic acid (Lab Scan) were used without further purification. The ITO coated PET 60 Ω was purchased from Sigma Aldrich.

3.1.2 Instruments

The synthesized polymer films were characterized for the electrochromic properties via the UV-visible spectrophotometer (Shimadzu, UV-1800). A Fourier transform infrared spectrometer (Thermo Nicolet, Nexus 670) was used for identify functional groups of PDMA synthesized in oxalic, nitric, and hydrochloric acids. A thermogravimetric analysis (PerkinElmer, Pyris Diamond TG/DTA) was utilized to investigate thermal properties of electropolymerized PDMA. The morphology of PDMA coated ITO surface was studied via FE-SEM (made, model?). The cyclic voltammetry (Multi-Channel Potentiostat VMP, Bio Logic Science Instrument) was used to study the oxidation-reduction properties of PDMA/ITO plastics.

3.2 Experimental Methods

3.2.1 Electropolymerization of PDMA on ITO plastic for the film appearance study

Poly (2,5-dimethoxy aniline) was electrodeposited onto a plastic ITO using potentials of 1.0 V and 1.5 V, in 25 ml 0.1 M 2,5-dimethoxy aniline in 0.1 M oxalic acid, hydrochloric acid, sulfuric acid, and nitric acid, and at various deposition times of 3, 5, 10, and 20 minutes. The stainless steel and plastic ITO were used as the counter and the working electrode, respectively.

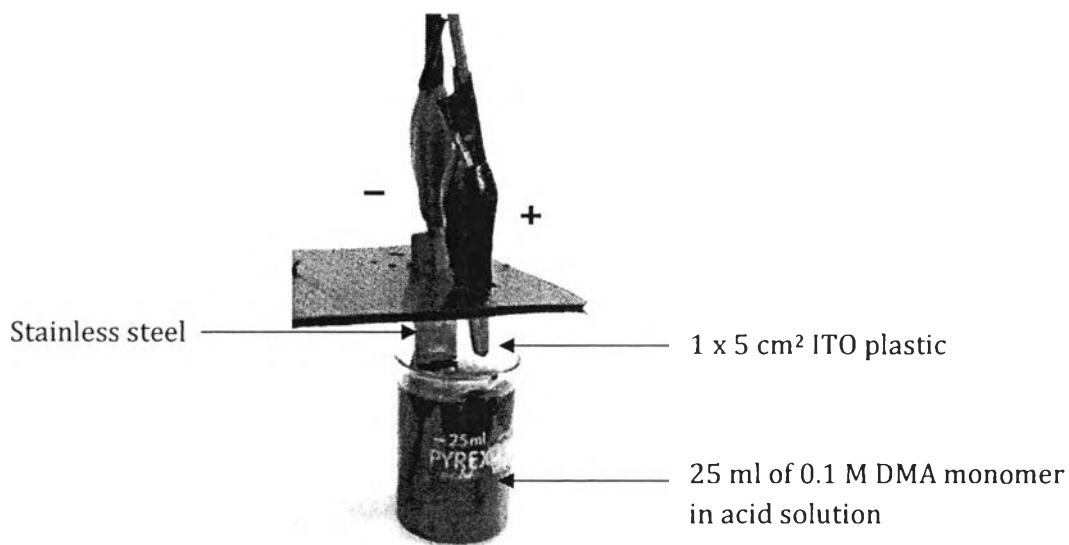


Figure 3.1 The experiment setup for the PDMA electropolymerization.

3.2.2 Electropolymerization of PDMA on ITO plastic for the electrochromic study

Poly (2,5-dimethoxy aniline) was electrodeposited onto a plastic ITO using potentials of 1.5 V, in 25 ml of 0.1 M 2,5-dimethoxy aniline in 0.1 M oxalic acid, hydrochloric acid, and nitric acid, and at various deposition times of 3, 6, and 10 minutes. The stainless steel and plastic ITO were used as the counter and the working electrode, respectively.

3.3 Characterization and Testing

3.3.1 Fourier Transform Infrared Spectrometer or FT-IR

The infrared absorption spectra of PDMA reveal the structure and functional groups of PDMA. A Fourier transform infrared spectrometer (Thermo Nicolet, Nexus 670) was used to identify the functional groups in the absorption mode with 64 scans, covering a wavenumbers range of $4000-400\text{ cm}^{-1}$, using a deuterated triglycine sulfate as a detector. Optical grade KBr was used as the background material.

3.3.2 Thermogravimetry Analysis or TGA

The thermogravimetric analyzer (Perkin Elmer, TGA7) was used to study the thermal stability and to determine the decomposition temperature of poly(2,5-dimethoxyaniline). The experiment was carried out by the electropolymerization of the DMA monomer in each acid solution on a stainless steel for 20 minutes. Then the PDMA coated stainless steels were dried and PDMA was scrapped into powder and kept at 105 °C for overnight before the TGA experiment. Measurements were carried out by weighting a powder sample of 2-3 mg and placed it in an alumina pan, and then heated it under nitrogen atmosphere with the heating rate of 10 °C/min from 50 - 900 °C.

3.3.3 UV-VIS Absorption Spectrophotometer

Optical absorption spectra as well as optical kinetic responses of the PDMA/ITO electrode polymerized from various acids were recorded on a Shimadzu spectroelectrometer UV-1800 (Figure 3.2). Measurements were carried out in a cuvette cell of 1 cm path length, assembled as an electrochemical cell with the PDMA film coated ITO plastic as the working electrode, the stainless steel wire as the counter electrode, and with the H₂SO₄ electrolyte solution as shown in Figure 3.3. The wavelength range scanned was 400-800 nm.

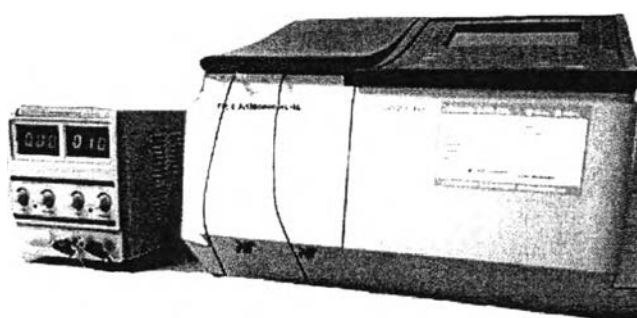


Figure 3.2 The UV–visible spectroelectrometer using for an electrochromic experiment.

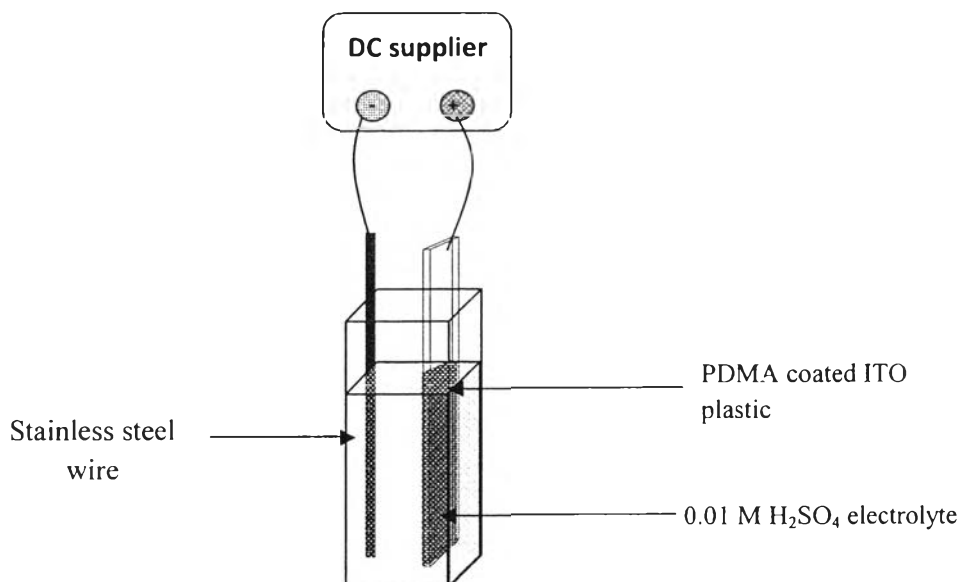


Figure 3.3 The schematic of PDMA electrochemical cell for electrochromic study.

3.3.4 Cyclic Voltammetry (CV) of PDMA

The PDMA/ITO electrodes synthesized in oxalic, nitric and hydrochloric acids at various dipping times (3, 6, and 10 minutes) were characterized for the redox properties by the cyclic voltammetry using a Multi-Channel Potentiostat (VMP, Bio Logic Science Instrument). Measurements were carried in 200 ml of 0.001 M H₂SO₄ background electrolyte, assembled with a three electrodes cell consisting of a saturated calomel electrode (SCE) as the reference electrode, a 5 cm length stainless steel wire as a counter electrode and 2 cm² PDMA/ ITO plastic was used as the working electrode, as shown in Figure 3.4.

The cyclic voltammogram (CV) of PDMA/ITO electrodes was measured in the potential range -1.0 to $+1.5$ V vs. SCE. The parameters and cell characteristics were setup as followed: the scan rate (dE/dt) of 10 mV/s, the data recorded every $dE_R = 1$ mV, and the electrode surface area of 2.00 cm². The electrochromic device was compiled with the following configuration:



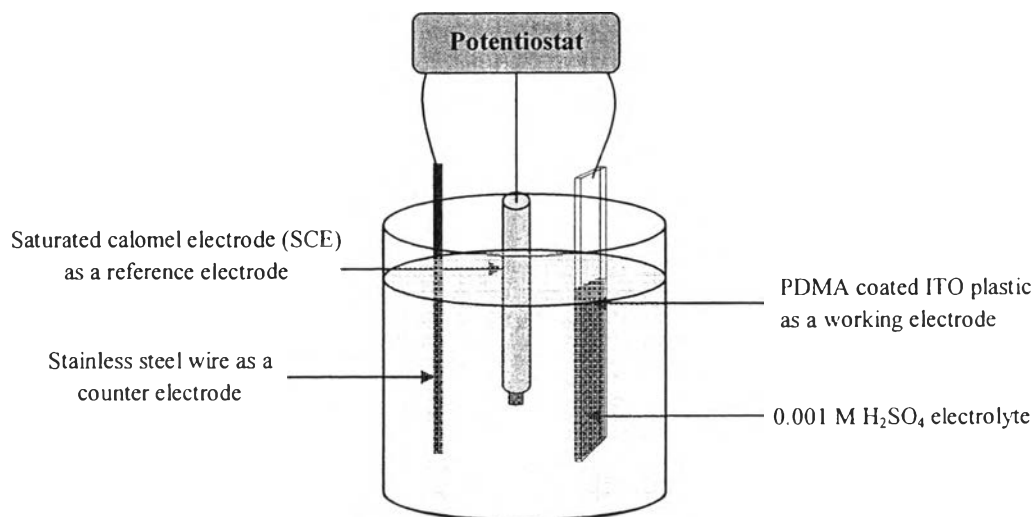


Figure 3.4 Schematic of a electrochemical cell of PDMA/ITO for the investigation of the redox properties via cyclic voltammetry.

3.3.5 Field-Emission Scanning Electron Microscopy

The field-emission scanning electron microscopy (HITACHI S4800) was used to determine the morphological structure and the surface appearance of PDMA synthesized with different acids and dipping times. The PDMA coated ITO was cut and then pasted on the carbon tape as shown in the Figure 3.5.

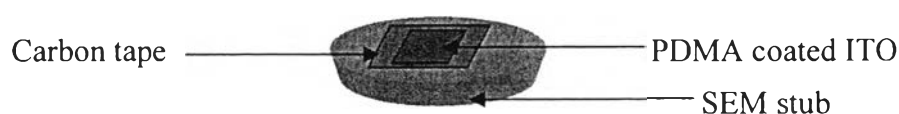


Figure 3.5 The schematic for the investigation of PDMA surface morphology via FE-SEM.