CHAPTER III
EXPERIMENTAL

3.1 Materials and Instruments

3.1.1 Materials

2,5-Dimethoxyaniline (Sigma Aldrich, AR grade, 98%) was used as the monomer. Oxalic acid (Ajax Finechem, AR grade) was used as the supporting electrolyte for the polymer synthesis. Hydrochloric acid (RCI Lab Scan, AR grade, 37%) and sulfuric acid (Lab Scan, AR grade, 98%) were used as electrolytes to investigate the electrochromic properties.

3.1.2 Instruments

The functional groups of PDMA were identified by a Fourier Transform Infrared spectrometer or FT-IR (Bruker, Equinox 55/FRA 106/S). The thermogravimetric analyzer (Perkin Elmer, TGA7) was used to study the thermal stability and determine the decomposition temperature of poly (2,5-dimethoxyaniline). The electrical conductivity of PDMA was measured by a two point probe couple with an electrometer (Keithley, model 6517A) under atmosphere condition. Multi-Channel Potentiostat (VMP, Bio Logic Science Instrument) was used to determine electrochemical properties of PDMA by the cyclic voltammetry. A UV-VIS absorption spectrophotometer (Shimadzu, UV-1800) was used to investigate the electrochromic properties, identify properties of conducting polymers related to electronic structure and optical behaviour upon the doping and the de-doping processes.

3.2 Experimental Methods : Synthesis of Poly(2,5-dimethoxyaniline) (PDMA)

Poly (2,5-dimethoxyaniline) was deposited as a conducting film on indium tin oxide (ITO) coated glass by the electrochemical polymerization (ECP) of 2,5-dimethoxyaniline monomer (DMA monomer) at a room temperature. The oxalic acid was used as the supporting electrolyte. A 0.125 mol of DMA was added in 0.1 M of oxalic acid. The electrochemical polymerization was carried out at 1.2 volt in a

cuvette of 1 cm path length with three electrochemical components: an ITO glass for coating PDMA film as the working electrode; another ITO glass as the counter electrode, and 0.1 M oxalic acid as supporting electrolyte as shown in Figure 3.1. Before each experiment, the ITO electrodes were cleaned with acetone and distilled water. The electrochromic device was compiled with the following configuration:

ITO || DMA – $C_2O_4H_2 \cdot 2H_2O$ || PDMA / ITO

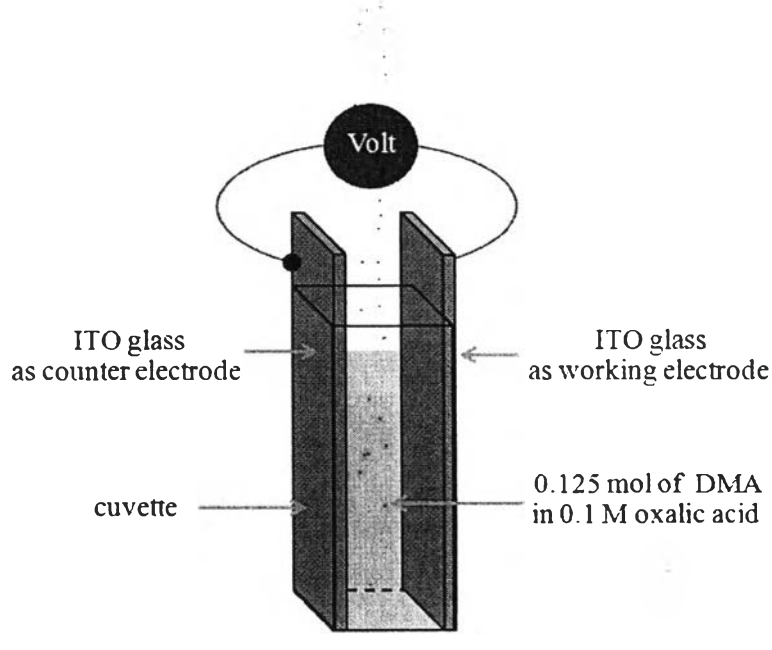

Figure 3.1 Schematic of a typical electropolymerization cell.

3.3 Characterization and Testing

3.3.1 Fourier Transform Infrared Spectrometer or FT-IR

FT-IR spectrometer (Bruker, Equinox 55/FRA 106/S) was used to characterize for the functional groups, in the absorption mode with 32 scans, with a resolution of ± 2 cm^{-1}, and in the wavenumbers range of 4000-400 cm^{-1}. Optical grade KBr (Carlo Erba Reagent) was used as the background material. The synthesized PDMA was mixed with dried KBr and pressed into a pellet form.

3.3.2 Two Point Probe Couple with an Electrometer

Two point probe couple with an electrometer (Keithley, model 6517A) was used to measure electrical conductivity under an atmosphere condition. A volatge was applied between probes number 1 and 2 and the resultant current was measured across the probes numbered 1 and 2. The specific conductivity, σ (S/cm) was obtained by measuring the resistance (R), and using the following relation

$$\sigma = \frac{1}{KRt} \qquad \text{Eq 3.1}$$

where t is the film thickness, and K is geometric correction factor or the ratio of the probe width divided by the probe length. A geometric correlation factor was calibrated by using standard silicon wafer sheets with known specific resistivity values. This measurement was repeated at least three times.

3.3.3 Thermalgravimetry Analysis or TGA

The thermogravimetric analyzer (Perkin Elmer, TGA7) was used to study the thermal stability and determine the decomposition temperature of poly (2,5-dimethoxyaniline). The experiment was carried out by weighting a powder sample of 5-10 mg, placed it in a platinum pan, and then heated it under a nitrogen flow with the heating rate 10 °C/min from 50 - 800 °C.

3.3.4 Cyclic Voltammetry (CV) of PDMA

Multi-Channel Potentiostat (VMP, Bio Logic Science Instrument) was used to determine electrochemical properties of PDMA by the cyclic voltammetry. Measurements were carried out in a cuvette of 1 cm path length with three electrodes: an Ag/AgCl electrode and two pieces of ITO glass used as the reference electrode, the working electrode, and the counter electrode, respectively. The potentials of CV were scanned between -0.5 to +0.5 V for 50 cycles with a sweep rate of 20 mV/s in a solution of 0.1 M of oxalic acid and 0.125 mol of DMA. In the the polymerization, a PDMA film deposited directly on the ITO electrode and was subjected to a potential scanning to obtain a stable cyclic voltammetric pattern (Huang et al., 2002).

3.3.5 UV-VIS absorption spectrophotometer

A UV-VIS absorption spectrophotometer (Shimadzu, UV-1800) was used to investigate the electrochromic properties, identify properties of conducting polymers related to electronic structure and optical behavior upon the doping and the dedoping processes. Measurements were carried out in a cuvette of 1 cm path length with three electrochemical components: with a PDMA film coated ITO glass as the working electrode; an ITO glass as the counter electrode, and 0.01 M H_2SO_4 or 0.01 M HCl as the supporting electrolyte. This spectrum was taken in the wavelength range of 300-900 nm, a scan speed of 240 mm/min, with a slit width of 1.0 nm, and using a deuterium and tungsten lamp as the light source.

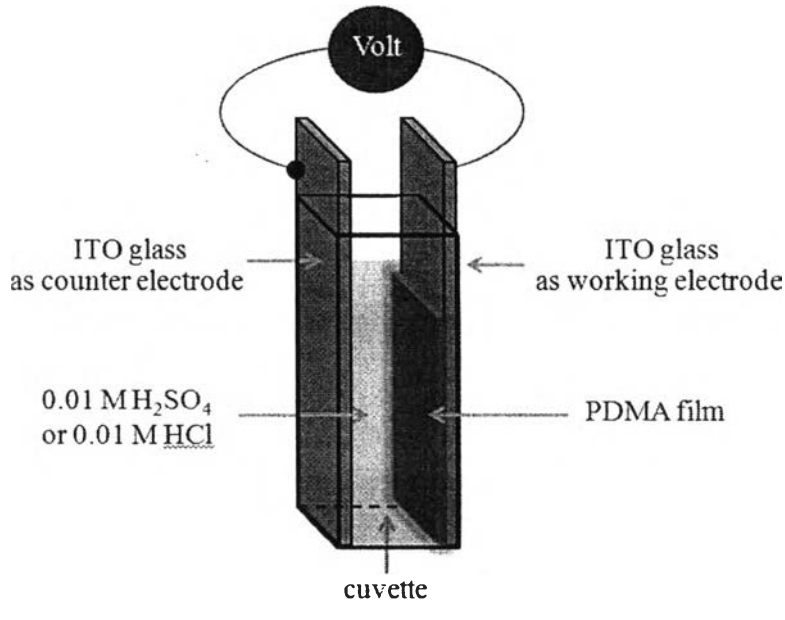

Figure 3.2 Schematic of a typical electrochemical cell.