

CHAPTER IV

MANUSCRIPT

Physical and Electrochromic Properties of Poly (2,5dimethoxy aniline) Synthesized in Oxalic, Nitric, and Hydrochloric Acids

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Abstract

One of the promising electrochromic polymers is polyaniline, which possess a high environmental stability and fast response time. Nonetheless, the low solubility in common organic solvents restricts processibility. A derivative of polyaniline, poly (2,5-dimethoxy aniline) or PDMA, shows higher solubility and a faster response time than polyaniline. In this work, poly (2,5-dimethoxy aniline) was electrochemically deposited on flexible indium tin oxide in oxalic, nitric and hydrochloric acids at various dipping times. Under different applied potentials, the optical contrast and response time of the PDMA coated plastic indium tin oxide were determined by a UV-visible spectroelectrometer. The fastest response times in all acids occurred at 3.5 volts, where the response time was only 3.7 seconds in case of 6 minutes synthesis of PDMA in hydrochloric acid. The Fourier transform infrared (FTIR) spectroscopy indicated that the structure of PDMA coating via three acids were nearly the same except that the FTIR spectrum of PDMA from nitric acid showed an absorption peak at 1384 cm⁻¹ representing N-O vibration. The thermal stability of the electropolymerized PDMA films was investigated via thermogravimetric analysis and showed similar three steps of weight loss at ~160 °C, 310 °C, and 450 °C. The surface morphology of PDMA coating depended on the type of acids used for the electropolymerization process.

Keywords: Electrochromic polymer, Poly (2, 5–dimethoxyaniline), Electrochemical polymerization

1. Introduction

Conductive polymers continue to have a growing interest because of the wide range of potential applications: optical display, rechargeable battery, electrochromic devices, and light emitting diodes [1]. Among organic molecules, the conductive polymers have attracted a significant interest in the field of electrochromism.

The electrochromism is the reversible and visible change in the light transmittance that is associated with an electrochemically induced oxidation-reduction reaction. The applications of such material are mirrors of vehicles [2], electrochromic displays [3], smart windows [4], earth-tone chameleon materials [5], ink-jet printers [6], and recently E-paper.

The earlier reports indicated using inorganic compounds or the metal oxides such tungsten trioxide (WO₃) and iridium dioxide (IrO₂) which are mix-valence metal complexes. However, these transition metal oxides electrochromically exhibited a slow response time (ten of seconds) with high processing cost. Compared with these materials, electrochromic polymers have shown several merits: low processing cost, power requirement only during switching, low operating voltage, great repeatability, no dependence with angle of vision, good UV stability, high coloration efficiency, fast switching ability, and ability for the fine-tuning of the band gap through the modification of polymer's chemical structure [2].

Potential conducting polymers are comprised of a number of functionalized polythiophenes, polypyrroles, polyanilines, and etc. They have become recognized as an interesting class of electrochromic materials [7]. An important issue in the study

of electrochromic polymeric materials has been that of controlling their colors by main-chain and pendant group structural modifications.

Polyaniline (PANI) is one of the most promising conducting polymers due to its straightforward polymerization and excellent chemical stability combined with relative high levels of conductivity [8]. Poly(2,5-dimethoxyaniline) or PDMA film showed reversible color changes from yellow to green representing the switching between the reduced state to the oxidized state through an application of potentials. Cyclic voltammetry results showed that PDMA can be easily switched from the leucoemeraldine state to the emeraldine state relative to PANI. PDMA also showed a shorter response time (9 s), which is smaller than the electrochemically prepared PANI (22 s) [9].

Thin-films can be created by several methods including the chemical vapor deposition, the electron beam deposition, the Langmuir–Blodgett technique, and the layer-by-layer (LbL) self-assembly technique [10]. In the case of electronically functional thin-films, thinner films are generally preferred because of the shorter electron and ion transport path length [11].

The present study aims to investigate the effect of the applied potentials and the electrolyte concentration on the electrochromic properties of PDMA electropolymerized on the ITO plastic via oxalic, nitric, and hydrochloric acids.

In this work, a 2,5-dimethoxyaniline (DMA) was polymerized via the electropolymerization with oxalic, nitric and hydrochloric acids with various dipping times (3, 6, and 10 minutes). The synthesized polymer films were characterized the

electrochromic properties via the UV-visible spectroelectrometer. The FTIR and TGA were utilized to investigate the structural and thermal properties of the electropolymerized PDMA. The morphology and thickness of PDMA coated ITO surface were studied via FE-SEM. The cyclic voltammetry was used to study the oxidation-reduction properties of PDMA/ITO plastic.

2. Experimental

2.1 Materials

2,5-dimethoxy aniline (DMA) (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.

2.2 The solubility test of 2,5-dimethoxy aniline monomer (DMA) in oxalic, hydrochloric, nitric, sulfuric, and acetic acids

The solution of 250 ml of 0.1 M acid was used to study the solubility of 0.1 M of 2,5-dimethoxy aniline monomer by sonication.

2.3 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 a counter and a working electrode, respectively.

2.4 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, hydrochloric, and nitric acids, and at various deposition times of 3, 6, and 10 minutes. The stainless steel and plastic ITO were used as a counter and a working electrode, respectively.

2.5 Characterization of PDMA/ITO film

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 in the absorption mode with 64 scans, covering a wavenumbers range of 4000-400 cm⁻¹. Optical grade KBr was used as the background material.

The thermogravimetric analyzer (Perkin Elmer, TGA7) was used to study the thermal stability and to determine the decomposition temperature of poly(2,5dimethoxyaniline). 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

I 28373248

then heated it under nitrogen atmosphere with the heating rate of 10 $^{\circ}$ C/min from 50 - 900 $^{\circ}$ C.

The PDMA/ITO electrodes synthesized in oxalic, nitric and hydrochloric acids at various dipping times (3, 6, and 10 minutes) were characterized 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 a reference electrode, a stainless steel wire as a counter electrode and 2 cm² PDMA/ ITO plastic was used as the working electrode. 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; scan rate (dE/dt) 10 mV/s, the data recorded every dE_R = 1 mV, and the electrode surface area 2.00 cm².

The field-emission scanning electron microscopy (HITACHI S4800) is used to determine the morphological structure of PDMA coated ITO via different acids and dipping times.

2.6 The Thickness of PDMA on ITO Substrate

The thicknesses of PDMA on ITO substrate deposited via oxalic, nitric, and hydrochloric acids for 3, 6, and 10 minutes were investigated by cross-sectional FE-SEM (HITACHI S4800) [12].

2.7 The Electrochromic Study of PDMA Coated ITO Plastics

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. 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. The wavelength range scanned was 400-800 nm. The kinetic absorption was observed at 750 nm [13] in the study of the effect of the applied potential on the response time which the potentials applied to the PDMA/ITO were 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 5.0, and 10 V in the 0.01 M sulfuric electrolyte solution. The effect of the electrolyte concentration on the response time was studied at applied potential 3.5 V in various concentrations of sulfuric acid $(10^{-6}, 10^{-5}, 10^{-4}, 10^{-3}, 10^{-2}, and 10^{-1} M)$. The

3. Results and Discussion

3.1 The Solubility of 2,5-Dimethoxy Aniline Monomer (DMA) in Oxalic,

Hydrochloric, Nitric, Sulfuric, and Acetic Acids

After 15 minutes of the sonication, 0.1 M (3.8295 g) 2,5-dimethoxy aniline monomer (DMA) was totally dissolved in 250 ml of oxalic, hydrochloric, nitric and sulfuric acids resulted in the deep black solution. However, after 30 minutes sonicating, the DMA monomer can partially dissolve in acetic acid.

Therefore, the electropolymerization of PDMA for the film appearance study was done in oxalic, hydrochloric, nitric and sulfuric acids.

3.2 Electropolymerization of PDMA on ITO Plastic for the Film Appearance Study

The electropolymerization of PDMA on ITO plastics in all acid at the potential 1.5 V offers a better PDMA film in which the film has a lesser gradient of color than 1.0 V. Moreover, the PDMA synthesized in 1.5 V results in a smoother surface with a shorter dipping time. On the other hand, the H_2SO_4 condition did not give the homogeneous film at both 1.0 V and 1.5 V.

3.3 FTIR Spectra of PDMA/ITO Film

The spectra of PDMA synthesized in oxalic, nitric, and hydrochloric acids are indicated in Fig. 4.1. The peak around 3400 cm⁻¹ can be attributed to the secondary N–H stretching modes for the amine functional group. The band at 1570 cm⁻¹ belongs to the C=N and C=C stretching modes for the benzenoid rings.

Quinoid and benzenoid peaks of PDMA ~1490 and 1580 cm⁻¹ are not clearly observed. This may be due to the overlapping with the aromatic C=C stretching bands ranging from 1400 to 1600 cm⁻¹ [14]. No vibration bands could be found in the range between 1700 and 2800 cm⁻¹, since no functional groups of PDMA can absorb in this region [14]. The weak band at ~1647 cm⁻¹ is indicative of the presence of the carbonyl group C=O contained in oxalate ion [15]. The FTIR spectrum of PDMA synthesized in nitric acid include an additional peak at 1384 cm⁻¹ corresponding to the nitrate group acting as a counter ion with the protonated amine [16].

These FTIR results suggest that the acid-doped PDMA films are in the emeraldine and pernigraniline forms, as both quinoid and benzenoid peaks appear in the spectra and the dark-green synthesized PDMA films suggest the dominance of the emeraldine and pernigraniline forms of the acid-doped PDMA films.

3.4 Thermogravimetric Analysis

The decomposition temperatures from the TGA curves indicate that PDMA synthesized in three acids decompose in the same three steps, as tabulated in Table 4.1 [17]. The initial weight loss at \sim 130°C is due to the loss of water bound molecules or moisture. The weight loss for PDMA at temperatures around 300 °C can be attributed to the removal of dopant anions [14]. The third weight loss at around 450°C indicates a structural decomposition of the polymer chain.

3.5 The Morphology of PDMA Surface

The morphology of PDMA is different when PDMA is synthesized in the different acids (Table 4.2) indicating that the type of acids plays an important role in directing the morphology and in aligning the monomers. PDMA from oxalic acid shows a high porosity microfiber structure [18] while the structure becomes more disordered microfiber when synthesized in nitric acid. The morphology of PDMA from hydrochloric acid is like the ordered tiny-granular aggregate. For all acids, as the polymerization continues, the surface morphology becomes more disordered along with particle aggregation.

3.6 The Thickness of PDMA on ITO Substrate

The thickness of PDMA films coated on ITO substrate via oxalic, nitric, and hydrochloric acids for 3, 6, and 10 minutes were investigated by cross-sectional FE-SEM. The result shows that the more polymerization time, the more thickness of PDMA film [19]. When the polymerization time increase from 3 to 10 minutes, the thickness of PDMA film increase from 109 ± 5 nm to 164 ± 8 nm, and 140 ± 5 nm to 188 ± 8 nm in case of oxalic acid and nitric acid, respectively. The thicknesses of PDMA from hydrochloric acid were 78 ± 3 nm and 104 ± 2 nm at 6 and 10 minutes deposition as shown in Table 4.3. The PDMA from hydrochloric acid shows the thinnest film among other acids at the same dipping time because of the slow polymerization rate. The slow polymerization rate is due to the lack of oxygen atom in acid molecule causing no oxygen linkage between the ITO surface and DMA monomer [15, 20].

3.7 The Absorption Spectrum of PDMA

The absorption spectra of PDMA film synthesized in oxalic, nitric, and hydrochloric acids were recorded at ± 1.0 V in the 0.01 M sulfuric acid. The spectrum of oxidation exhibits the band at $\lambda_{max} \sim 750$ nm corresponds to pernigraniline form. At -1.0 V, the spectrum shows an absorption band at $\lambda_{max} = 480$ nm correspond to the reduced state (leucoemeraldine) which disappears upon an oxidation.

3.8 The Kinetic Coloration of PDMA

3.8.1 The effect of the testing potentials on the response time

The study of the kinetic coloration of PDMA coated ITO electrode was carried out via the kinetic mode of UV-visible spectroelectrometer. The wavelength 750 nm representing the oxidized state was used to study the response time and optical contrast between the redox states. The measurement was set to collect the absorbance data in every second. The potentials applied to the PDMA/ITO were 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 5.0, and 10 V.

The applied potential of -0.5 V, the PDMA films from all acids showed a very slow reduction (response time \sim 1 hr) contributing to the corrosion of the PDMA/ITO films. Moreover, the applied potential of 4.0 V, 5.0 V, and 10 V are the overvoltage of the PDMA switching which the PDMA shows the irreversible color change [21]. Accordingly, the response time can study in 1.0 V to 3.5 V potentials range.

The effect of the testing potentials on the reduction time, oxidation time, and response time of PDMA synthesized in oxalic acid at various dipping times are shown in Figs. 4.2-4.4. When the potential increase from 1.0 V to 3.5 V, the reduction time decrease from 110 s. to 6.8 s., 110 s. to 9.0 s., and 210 s. to 11.5 s. for 3, 6, and 10 minute deposition time. When the potential increase from 1.0 V to 3.5 V, the oxidation time decrease from 170 s. to 6.0 s., 180 s. to 12 s., and 175 s. to 14 s. for 3, 6, and 10 minute deposition time.

The results suggest that, for all acid, when the potential increase, the response time decrease. At 6 minutes dipping time, the response times of PDMA decrease from 290 s. to 21 s. (oxalic acid), 515 s. to 38 s. (nitric acid), and 182 s. to 3.7 s. (hydrochloric acid) when the applied potentials increase from 1.0 V to 3.5 V in 0.01 M H₂SO₄ electrolyte. At 10 minutes dipping time, the response times of PDMA

decrease from 385 s. to 25.5 s. (oxalic acid), 1310 s. to 35 s. (nitric acid), and 350 s. to 15 s. (hydrochloric acid) when the applied potentials increase from 1.0 V to 3.5 V.

The fastest response time of PDMA from oxalic acid is 12.8 s. at 3 minutes synthesis with 3.5 V testing potential. The slowest response time of PDMA from oxalic acid is 385 s. from the 10 minutes synthesis with testing potential 1.0 V.

The fastest response time of PDMA from nitric acid is 20 s. at 3 minutes synthesis with 3.5 V testing potential. The slowest response time of PDMA from oxalic acid is 1310 s. from the 10 minutes synthesis with testing potential 1.0 V.

The fastest response time of PDMA from hydrochloric acid is 3.7 s. at 6 minutes synthesis with 3.5 V testing potential. The slowest response time of PDMA from oxalic acid is 350 s. from the 10 minutes synthesis with testing potential 1.0 V.

The results of the response time of PDMA from various acids indicate that the increase of polymerization time contributes to the increase of the response time since the increase in the thickness of the PDMA film. Moreover, the PDMA from hydrochloric acid shows the fastest response time (3.7 s.) in all acid at the same dipping time because the thinnest film from the slow polymerization rate in hydrochloric acid. The response time 3.7 s. is faster than that of the device ITO/PDMA || LiClO₄-PC-PEO (400,000)||WO₃/ITO [9] since liquid electrolyte has higher ion mobility than gel electrolyte. 3.8.2 The effect of the electrolyte concentration on the response time

The response time at 3.5 V is fastest for all acids, so this potential was used to study the effect of the concentration on response time of PDMA. The films of PDMA synthesized in oxalic and nitric acids for 3 minutes and in hydrochloric acid for 6 minutes show the fastest response time among other dipping times, hence these films were further used to study the effect of electrolyte concentration.

The PDMA coated ITO plastics with various acids were tested at 3.5 V in various concentrations of sulfuric acid $(10^{-6}, 10^{-5}, 10^{-4}, 10^{-3}, 10^{-2}, \text{ and } 10^{-1} \text{ M})$. Fig. 4.5 indicates that the increase of the electrolyte concentration from 10^{-6} to 10^{-2} M result in the reduction the response times from 460 s. to 13.5 s., 760 s. to 36 s., and 160 s. to 3.7 s. for PDMA synthesized in oxalic, nitric, and hydrochloric acids, respectively.

3.8.3 The effect of the type of acids and the dipping time on the optical contrast

The high optical contrast is one of the important properties of the efficient electrochromic polymer. In the study, the optical contrast (Δ Abs) of PDMA/ITO was determined as the absorbance difference between the steady state value of the reduction and oxidation states. The optical contrast of PDMA coated ITO plastics via oxalic, nitric, and hydrochloric acids at 6 and 10 minutes are shown in Table 4.4. The optical contrast of the PDMA films slightly increase when the polymerization time increase from 6 to 10 minutes. PDMA synthesized with the same dipping time shows the optical contrast in the order of nitric acid > oxalic acid

> hydrochloric acid. This is the evidence that the optical contrast depends on thickness of the PDMA film. The highest contrast between the bleached and the colored forms is observed at λ =750 nm, (Δ T%=73%) which higher than that of ITO/PDMA electrode (Δ T%=23%) [9].

3.9 Cyclic Voltammetry (CV) of PDMA

The PDMA deposited ITO via oxalic, nitric, and hydrochloric acids show two main redox processes in which the first weak indicates the oxidation at around -0.5 V related to the leucoemeraldine/ emeraldine (yellow to green) transformation, and the second sharp peak corresponds to the emeraldine/ pernigraniline (green to blue) transformation as same as the previous studies [22,23].

Cyclic voltammograms (CV) of PDMA/ITO synthesized in oxalic acid at various times are shown in Fig. 4.6 respectively. The peak potentials (E_{pc} , E_{pa}) and the peak currents (I_{pc} , I_{pa}) of the cathodic and anodic peaks are tabulated in Table 4.5. In case of PDMA from oxalic acid, the I_{pc} and I_{pa} increase from 0.18 mA and 0.5 mA to 0.44 mA and 0.68 mA, respectively when the polymerization time are raised from 3 to 10 minutes. The increase of the current by the increase of the polymerization time can be seen in PDMA from nitric and hydrochloric acids, as well, due to the increase of mass on the electrode.

When the polymerization times increase, the oxidation-reduction potentials of PDMA from all acids are shifted to being more positive and negative, respectively, because of the greater diffusion time of the counter ion for the redox reaction through the thicker films.

4. Conclusions

The PDMA synthesized shows the reversible color change from yellow to green representing the transition between the leucoemeraldine (reduced state) and emeraldine (oxidized state) upon the switching potentials. The PDMA/ITO electropolymerized in oxalic, nitric, and hydrochloric acids have mostly the same structural property, the only difference is the FTIR band of the oxalate and nitrate groups acting as counter ions with the protonated amine. The decomposition temperatures from the TGA curves of the PDMA synthesized in three acids occur at three steps: ~130 °C, 300 °C, and 450 °C. The important differences of the PDMA synthesized in various acids are the morphology and the thickness of PDMA films at the same dipping time, both factors lead to the variations of the response time and the optical contrast.

The results from the UV-Visible spectroelectrometer indicate that the increase of the applied potentials (1.0 V to 3.5 V) and the electrolyte concentration $(10^{-6} \text{ M to } 10^{-1} \text{ M})$ contribute to the abruptly decrease of the response time. The cyclic voltammetry shows the shift of the oxidation-reduction potentials of PDMA from all acids to being more positive and negative, respectively with the increase of the polymerization times because of the greater diffusion time of the counter ion for the redox reaction through the thicker films.

Acknowledgements

The authors would like to acknowledge the financial supports from the Conductive and Electroactive Research Unit of Chulalongkorn University, the Center of Excellence on Petrochemical and Materials Technology, the Thailand Research Fund (TRF-BRG), Nanotec, and the Royal Thai Government.

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Figures Caption

Fig. 4.1 FTIR spectra of PDMA electropolymerized in oxalic, hydrochloric and nitric acids.

Fig. 4.2 The effect of the testing potentials on the reduction time of PDMA synthesized in oxalic acid at various dipping times.

Fig. 4.3 The effect of the testing potentials on the oxidation time of PDMA synthesized in oxalic acid at various dipping times.

Fig. 4.4 The effect of the testing potentials on the response time of PDMA synthesized in oxalic acid at various dipping times.

Fig. 4.5 The effect of H_2SO_4 concentration on the response time at 3.5 V of PDMA coated ITO electropolymerized in each acid.

Fig. 4.6 Cyclic voltammograms of PDMA/ITO synthesized in oxalic acid at 3, 6, and 10 minutes.

Table Captions

Table 4.1 Three weight loss steps of PDMA polymerized via the three acids Table 4.2 Surface Morphology of PDMA polymerized for 6 and 10 minutes via various acids

Table 4.3 Thickness of PDMA/ITO synthesized in various acids at 3, 6, and 10 minutes dipping time

Table 4.4 The optical contrast of PDMA coated ITO plastic via nitric, oxalic and hydrochloric acids for 6 and 10 minutes

Table 4.5 The peak potentials (E_{pc}, E_{pa}) and peak currents (I_{pc}, I_{pa}) of the PDMA synthesized in oxalic, nitric, and hydrochloric acids



Fig. 4.1 FTIR spectra of PDMA electropolymerized in oxalic, hydrochloric and nitric acids.



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Fig. 4.3 The effect of the testing potentials on the oxidation time of PDMA synthesized in oxalic acid at various dipping times.



Fig. 4.4 The effect of the testing potentials on the response time of PDMA synthesized in oxalic acid at various dipping times.



Fig. 4.5 The effect of H_2SO_4 concentration on the response time at 3.5 V of PDMA coated ITO electropolymerized in each acid.



Fig. 4.6 Cyclic voltammograms of PDMA/ITO synthesized in oxalic acid at 3, 6, and 10 minutes.

Acids for the	First loss (°C)	Second loss (^{0}C)	Third loss (^{0}C)
electropolymerization	1 113(1035 (C)		Third loss (°C)
Oxalic acid	164	306	475
Hydrochloric acid	163	307	448
Nitric acid	160	312	432

Table 4.1 Three weight loss steps of PDMA polymerized via the three acids

various acids

Acids	Dipping time			
1 total	6 min	10 min		
Oxalic acid	500 nm	<u>500 пт</u>		
Hydrochloric acid	500 nm	<u>500 nm</u>		



 Table 4.3 Thickness of PDMA/ITO synthesized in various acids at 3, 6, and 10

 minutes dipping time

Acids	Thickness (nm)		
Acids	3 min	6 min	10 min
Oxalic acid	109 ± 5	144 ± 5	164 ± 8
Nitric acid	140 ± 5	166 ± 9	188 ± 8
Hydrochloric acid	No deposition	78 ± 3	104 ± 2

Table 4.4 The optical contrast of PDMA coated ITO plastic via nitric, oxalic and

 hydrochloric acids for 6 and 10 minutes

Type of acids	Polymerization time		
Type of delus	6 min	10 min	
Nitric acid	2.260 ± 0.360	2.330 ± 0.300	
Oxalic acid	1.770 ± 0.240	2.010 ± 0.140	
Hydrochloric acid	0.372 ± 0.097	1.170 ± 0.440	

Acids	Polymerization	Current (mA)		Potential (V)	
	time (minutes)	I _{pc}	I _{pa}	E _{pc}	E _{pa}
Oxalic acid	3	0.18	0.5	-0.07	0.5
	6	0.28	0.64	-0.08	0.7
	10	0.44	0.68	-0.32	0.8
Nitric acid	3	0.52	0.52	-0.35	0.7
	6	0.72	0.74	-0.5	0.8
	10	0.9	0.85	-0.8	1.1
Hydrochloric	6	0.3	0.3	-0.3	0.5
acid	10	4.7	4.7	-0.82	0.98

Table 4.5 The peak potentials (E_{pc}, E_{pa}) and peak currents (I_{pc}, I_{pa}) of the PDMA synthesized in oxalic, nitric, and hydrochloric acids