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APPENDICES

Appendix A Solubility

2,5-dimethoxy aniline monomer (DMA) was dissolved in some acids by sonicating but DMA did not dissolve in some acids, as shown in Table A1.

Table A1 Solubility of 0.1 M PDMA in 0.1 M of acids

Acids \ Sonicate time	15 min	30 min
Oxalic acid	dissolved	
Hydrochloric acid	dissolved	
Sulfuric acid	dissolved	
Nitric acid	dissolved	
Acetic acid		partially dissolved

Appendix B Electropolymerization

Poly (2,5-dimethoxy aniline) was electro-deposited onto a plastic ITO using potentials of 1.0 V and 1.5 V, in 25 ml of 0.1 M oxalic acid, hydrochloric acid, sulfuric acid, and nitric acid, and at various deposition times of 3, 5, 10, and 20 min. The experiment setup of the electropolymerization is shown in Figure B1. The results of PDMA synthesis are shown in Table B1.

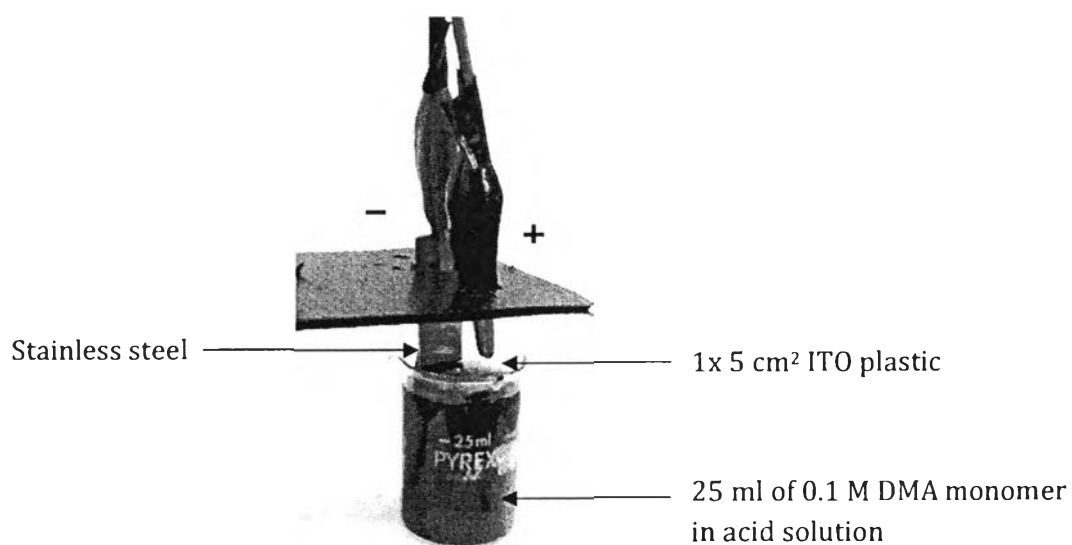


Figure B1 The experiment setup for the PDMA electropolymerization.

Table B1 The electropolymerization of PDMA on a plastic ITO

Acids	Voltage (V)	Deposition Time (min)			
		3	5	10	20
Oxalic acid	1.0	+	+	+	+
	1.5	++	++	++	++
HCl	1.0	-	-	++	++
	1.5	-	+	++	++
H ₂ SO ₄	1.0	-	-	-	-
	1.5	+	+	+	+
HNO ₃	1.0	-	++	++	++
	1.5	++	++	++	++

where - represents none of PDMA deposited.
 + represents some deposition of PDMA or gradient color
 ++ represents good deposition of PDMA (homogeneous color)

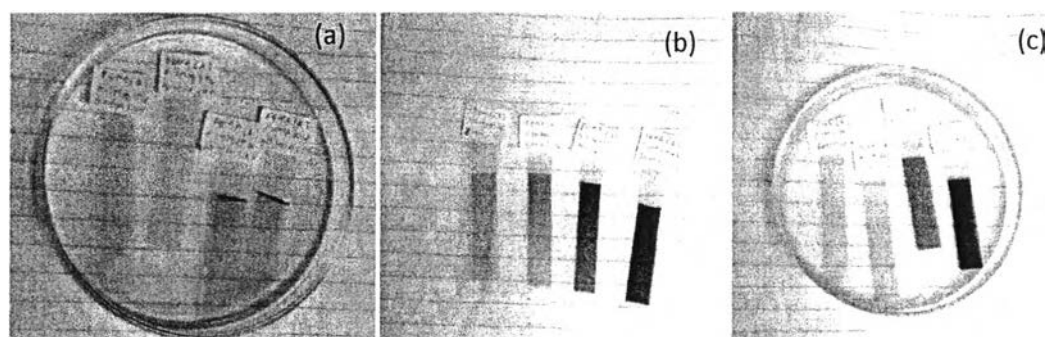


Figure B2 The PDMA synthesized on a ITO plastic with the dipping times of 3, 5, 10 and 20 min in 0.1 M H_2SO_4 at 1.0 V (a), 0.1 M oxalic acid at 1.0 V (b), and 0.1M HCl at 1.0 V(c).

Electropolymerization of PDMA 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 as shown in Figure B2. 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 was not chosen because the electropolymerization of PDMA in H_2SO_4 did not give the homogeneous film at both 1.0 V and 1.5 V.

Appendix C Identification of FTIR Spectra

The infrared absorption spectra of PDMA reveal the structure and functional groups of PDMA. FT-IR spectrometer (Bruker, Equinox 55/FRA 106/S) was used to identify the functional groups in the absorption mode with 64 scans, covering a wavenumbers range of 4000-400 cm^{-1} , using a deuterated triglycine sulfate as a detector. Optical grade KBr was used as the background material. FTIR spectra of PDMA polymerized via oxalic, hydrochloric, and nitric acids are shown in Figure C1 and the data are given in Table C1.

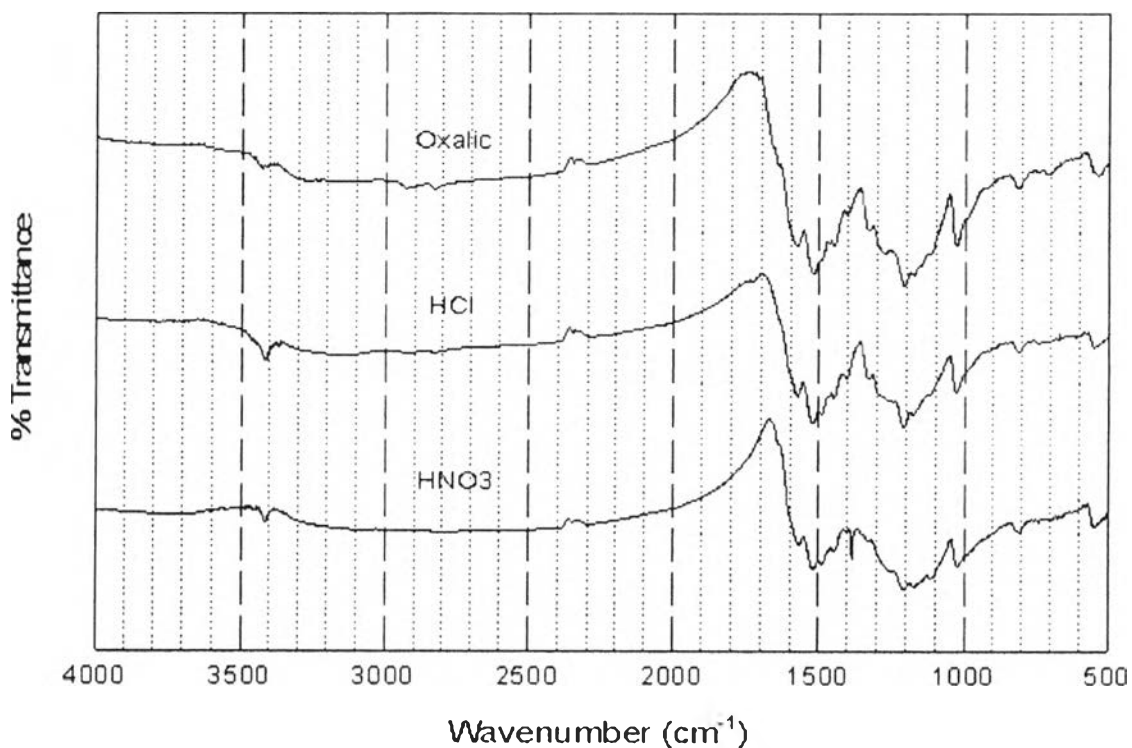


Figure C1 FTIR spectra of PDMA electropolymerized in oxalic , hydrochloric and nitric acids.

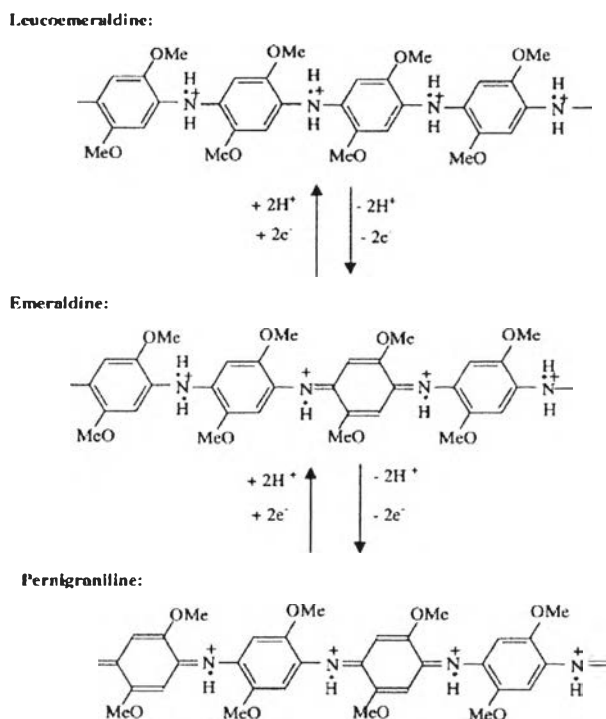


Figure C2 The structures of poly(2,5-dimethoxy aniline) under applied potential (Huang *et al.*, 2002).

Table C1 FTIR band assignments for the PDMA synthesized in three acids

Assignment	Oxalic acid	Hydrochloric acid	Nitric acid	References
N–H stretching	3422	3417	3416	Neelgund <i>et al.</i> , 2008
C=N and C=C stretching modes for the benzenoid rings.	1571	1571	1568	Mavundla <i>et al.</i> , 2008
C=C stretching modes for the quinoid rings.	1485	1493	1490	Mavundla <i>et al.</i> , 2008
Benzenoid C–N stretching	1518	1522	1518	Ragupathy <i>et al.</i> , 2008
carbonyl group stretching	1641	-	-	Patil <i>et al.</i> , 2004
N–O stretching	-	-	1384	Blinova <i>et al.</i> , 2009
Stretching of methoxy group	1205	1209	1205	Patil <i>et al.</i> , 2004

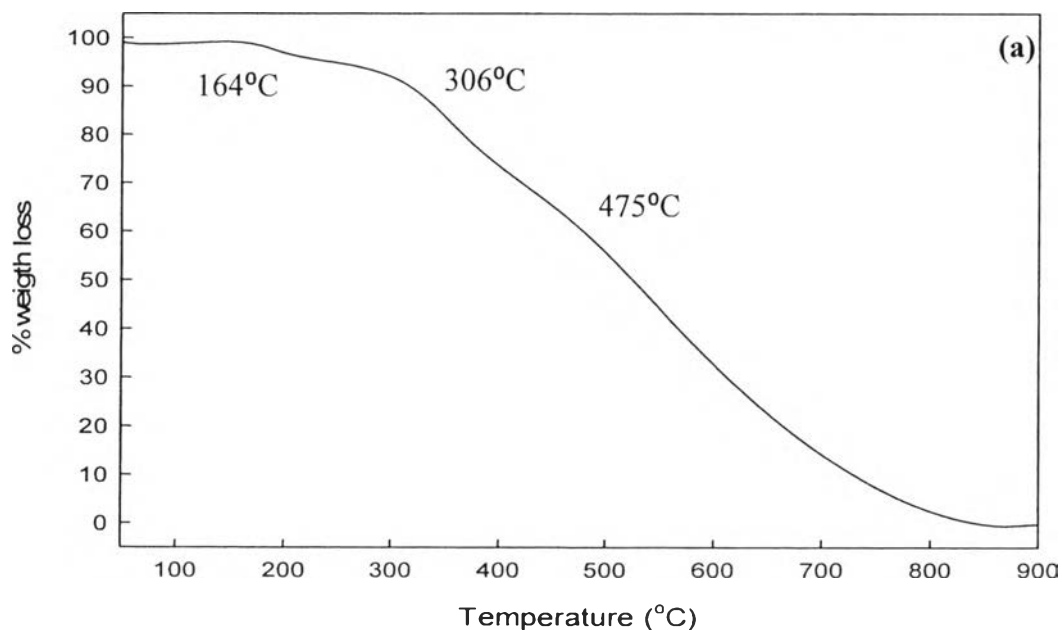
The spectra of PDMA synthesized in oxalic, nitric, and hydrochloric acids exhibit the characteristic bands in the 400 – 4000 cm^{-1} range. The peak around 3400 cm^{-1} can be attributed to the secondary N–H stretching modes for the amine functional group. The band at 1570 cm^{-1} belongs to the C=N 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} (Neelgund *et al.*, 2008). 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 (Neelgund *et al.*, 2008). The weak band at $\sim 1647 \text{ cm}^{-1}$ is indicative of the presence of the carbonyl group C=O contained in oxalate ion (Patil *et al.*, 2004). The FTIR spectrum of PDMA synthesized in nitric acid shows a peak at 1384 cm^{-1} , which is due to the nitrate group acting as a counter ion with the protonated amine.

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.

Appendix D Thermogravimetric 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. Thermogravimetric curves for the PDMA polymerized in oxalic, nitric and hydrochloric acids are given in Figure D1.



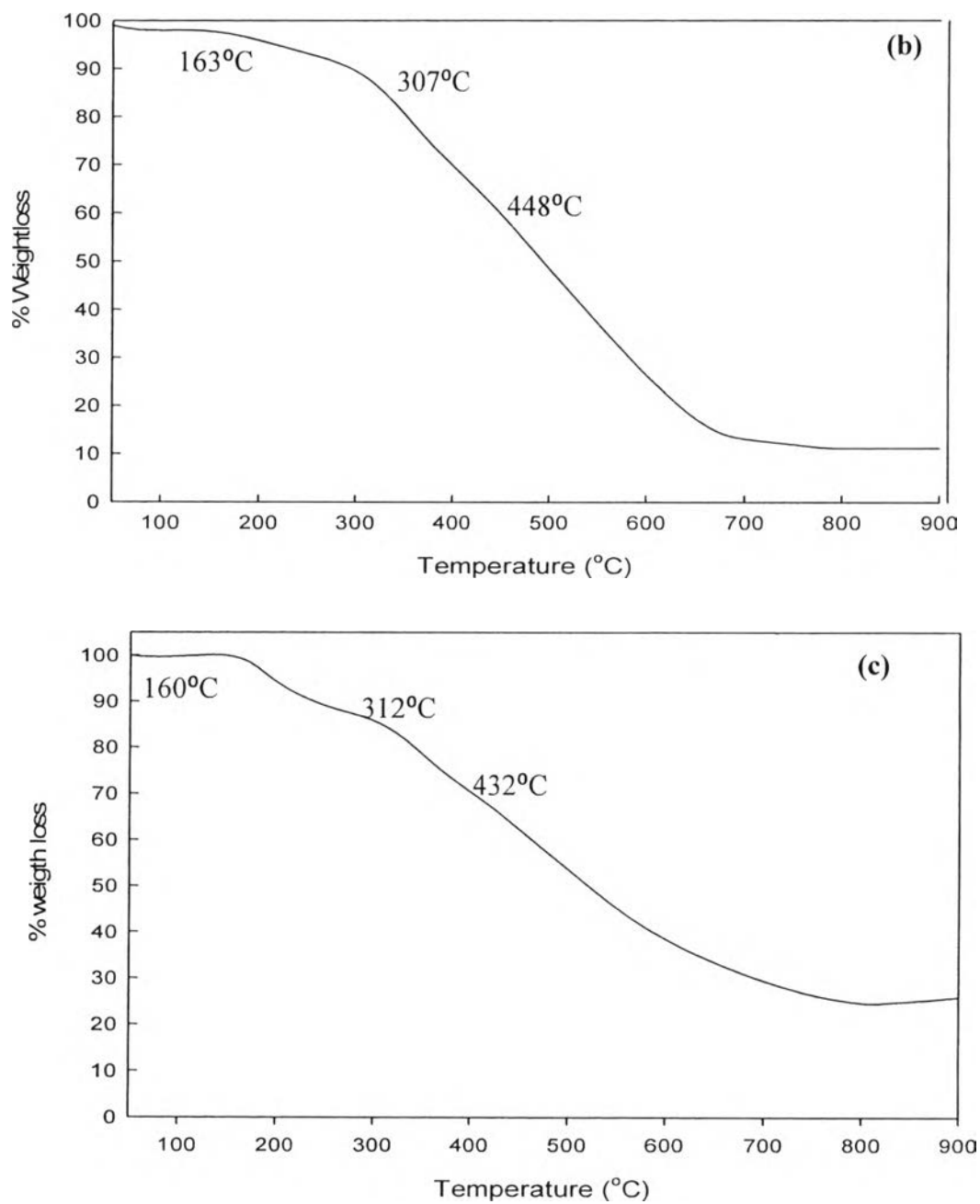


Figure D1 TGA curve for PDMA synthesized in: (a) oxalic acid; (b) hydrochloric acid; and (c) nitric acid.

Table D1 Three weight loss steps of PDMA polymerized via the three acids

Acids for the electropolymerization	First loss (°C)	Second loss(°C)	Third loss (°C)
Oxalic acid	164	306	475
Hydrochloric acid	163	307	448
Nitric acid	160	312	432

The decomposition temperatures from the TGA curves indicate that PDMA synthesized in three acid decompose in three steps (Mavundla *et al.*, 2008). The initial weight loss at $\sim 130^{\circ}\text{C}$ 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 (Neelgund *et al.*, 2008). The third weight loss at around 450°C indicates a structural decomposition of the polymer chain.

Appendix E The Electrochromic properties 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 spectrophotometer UV-1800 (Figure E1). 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 H_2SO_4 electrolyte solution as shown in Figure E2. The wavelength range scanned was 400-800 nm.

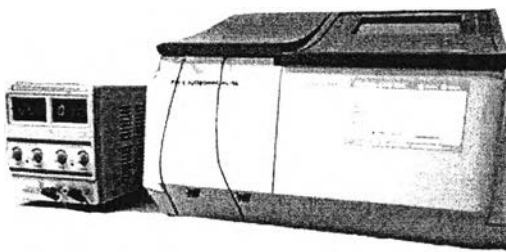


Figure E1 The UV–visible spectrophotometer using for an electrochromic experiment.

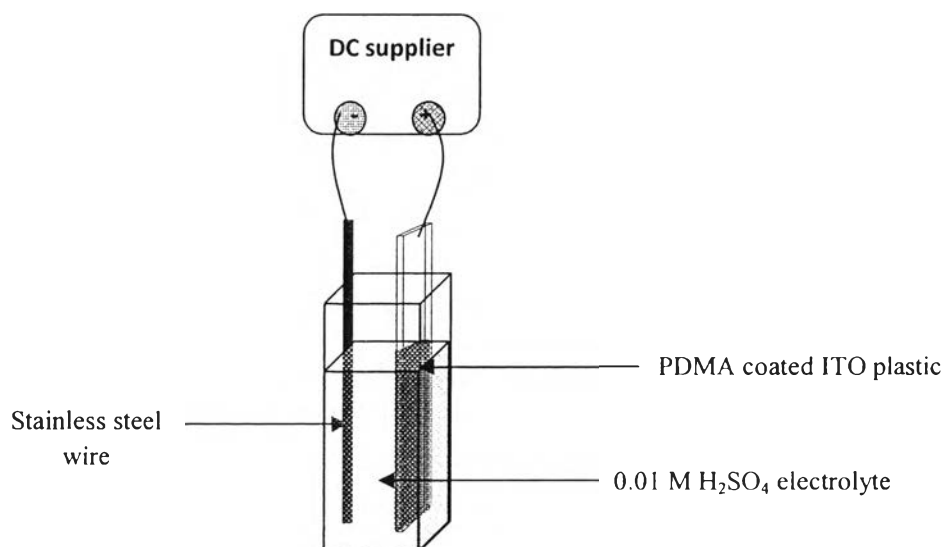


Figure E2 The schematic of PDMA electrochemical cell for electrochromic study.

The absorption spectra of PDMA film synthesized in oxalic, nitric, and hydrochloric acids recorded at 1.0 V with 2 minutes interval in the 0.01 M sulfuric acid show two absorption bands as shown in Figure E3, E4, and E5, respectively. The band at $\lambda_{\max} = 480$ nm correspond to the reduced state (leucoemeraldine). This band disappears upon an oxidation. The band at $\lambda_{\max} \sim 750$ nm corresponds to the oxidation state of PDMA or pernigraniline form.

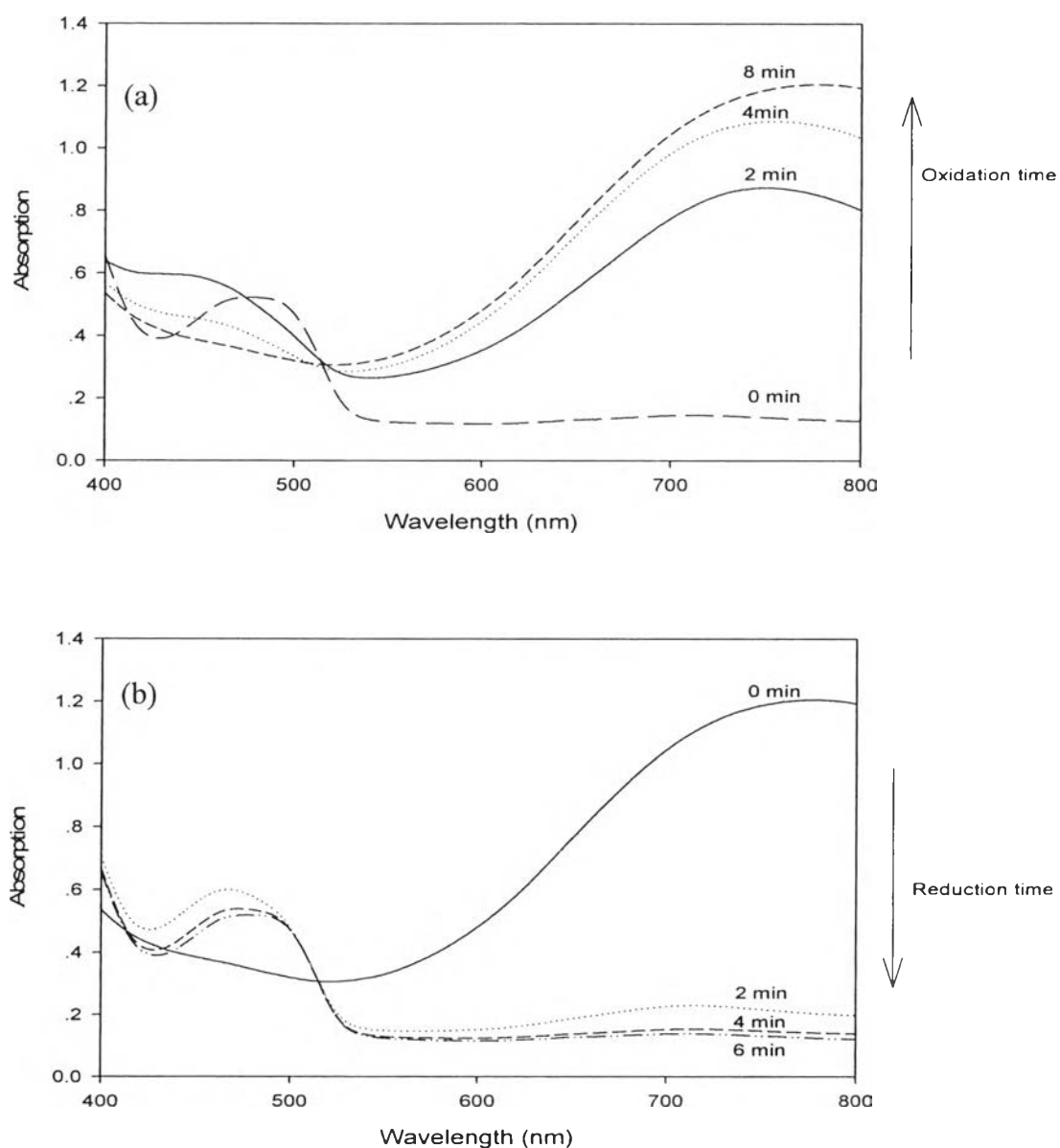


Figure E3 The absorption spectra of PDMA coated ITO plastic via oxalic acid for 3 minutes at (a) oxidized state applied potential of +1.0 V and (b) reduced state applied potential of -1.0 V.

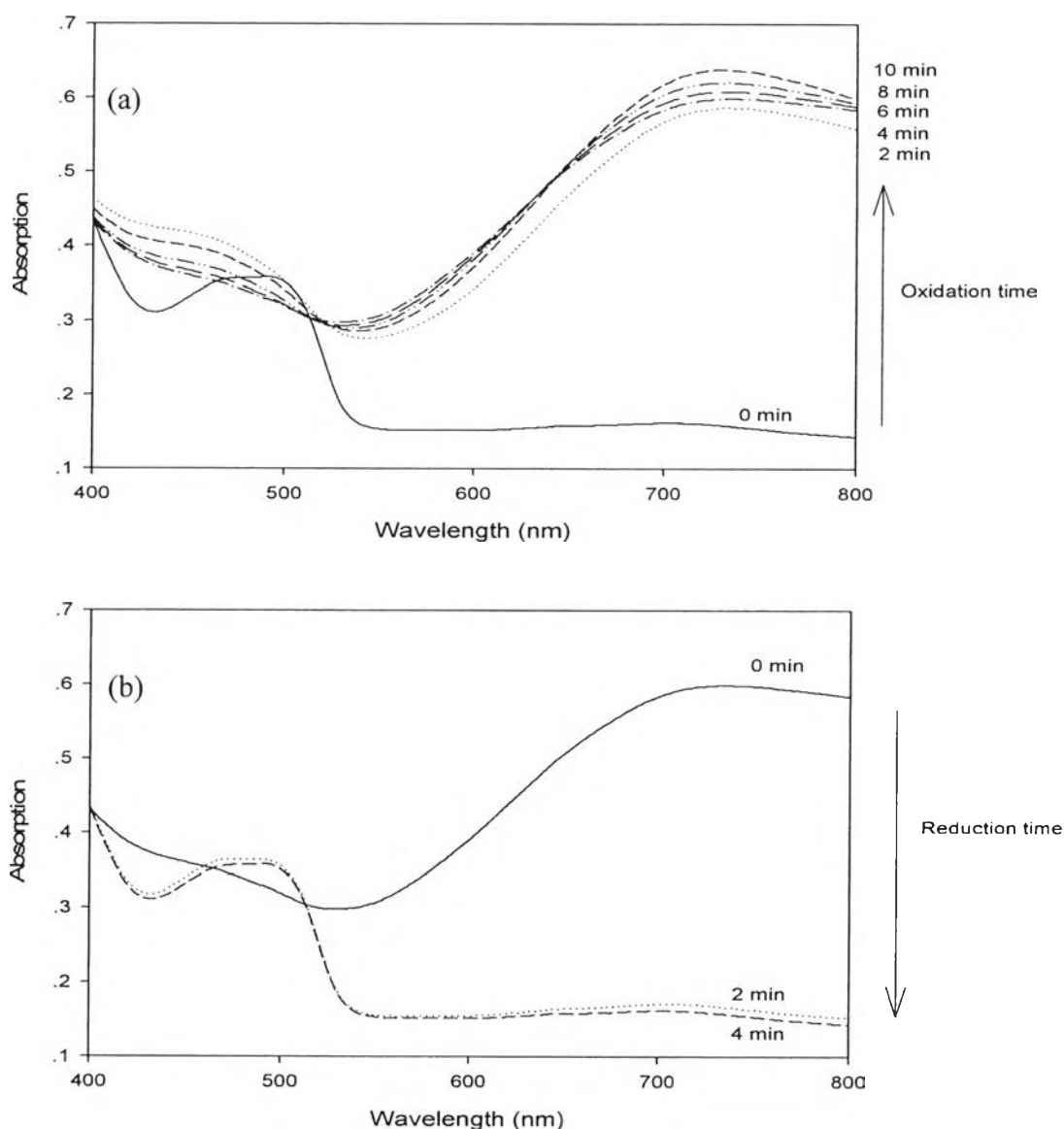


Figure E4 The absorption spectra of PDMA coated ITO plastic via nitric acid for 3 minutes at (a) oxidized state applied potential of +1.0 V and (b) reduced state applied potential of -1.0 V.

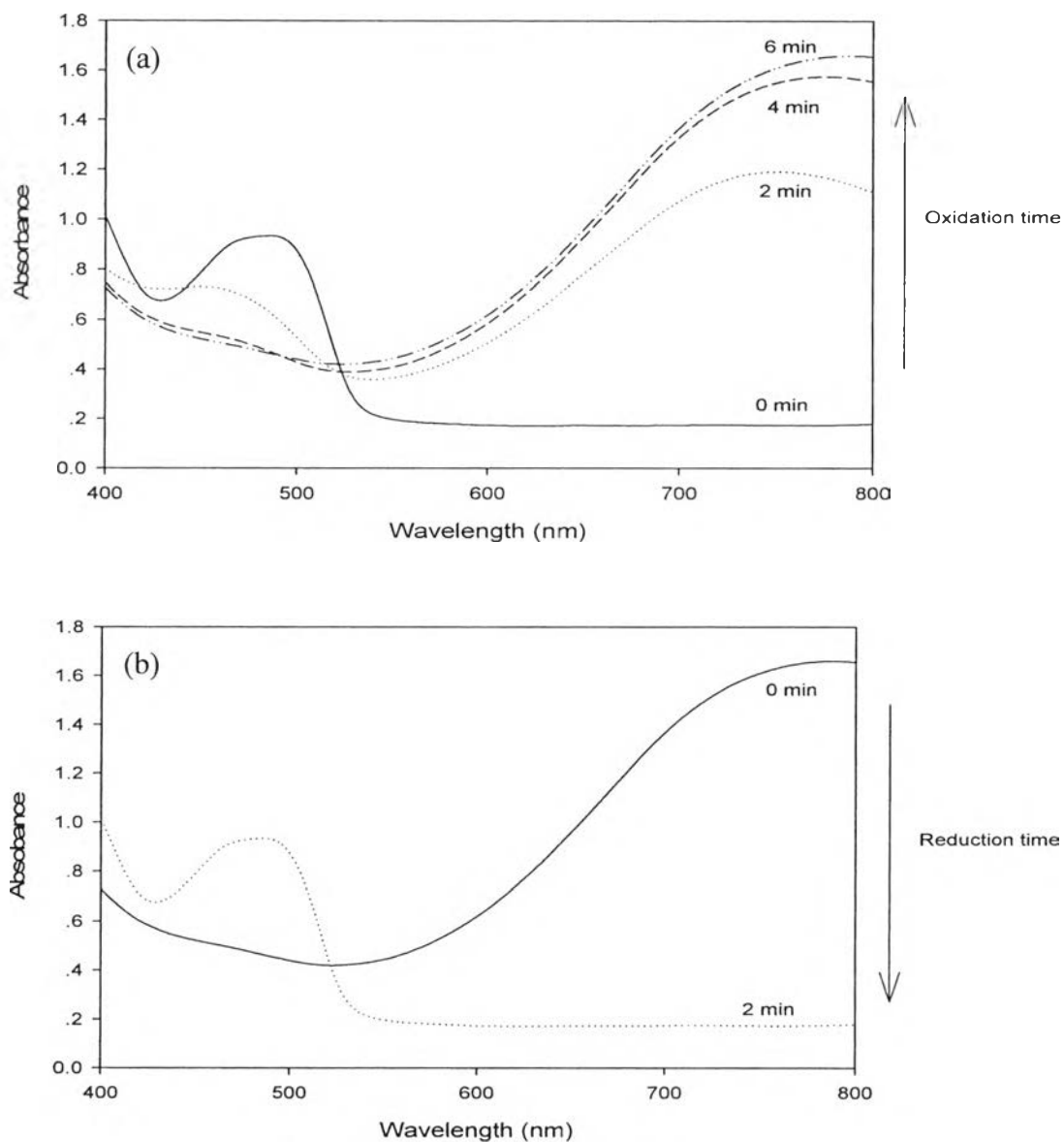
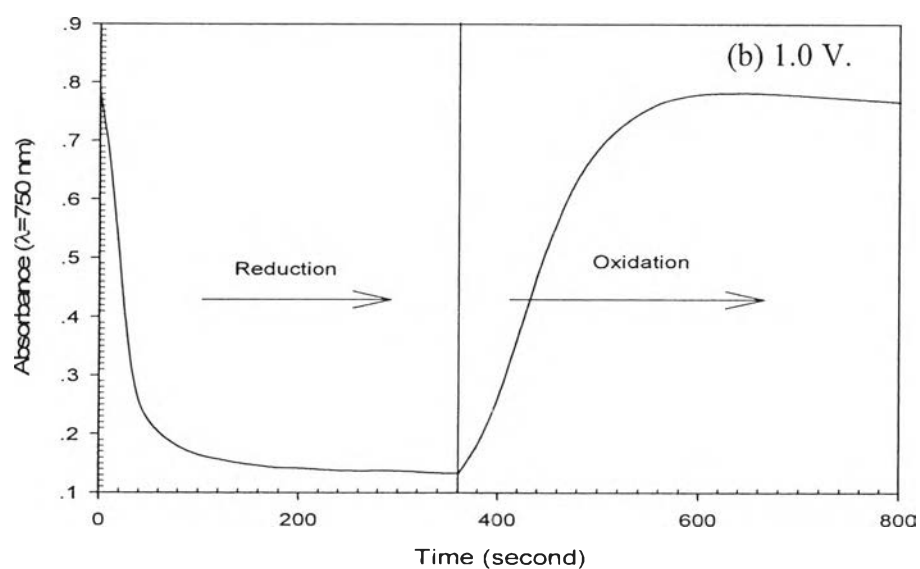
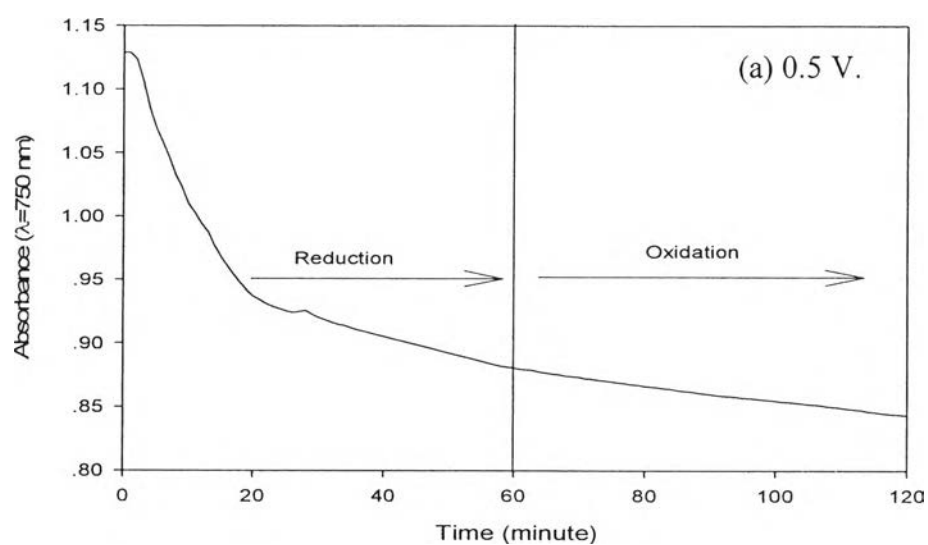


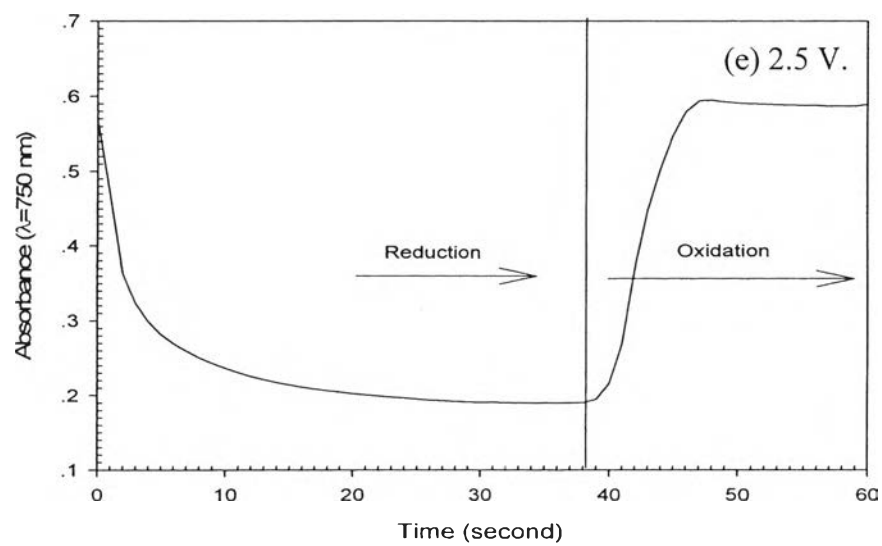
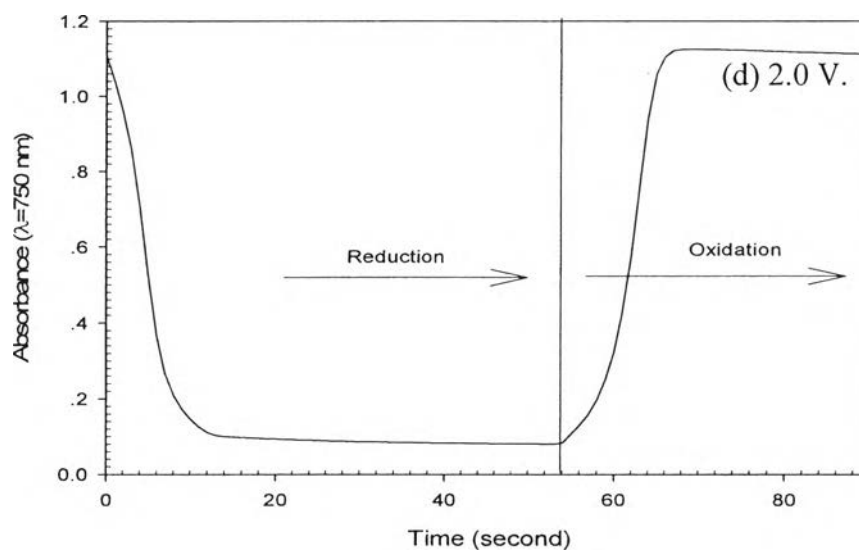
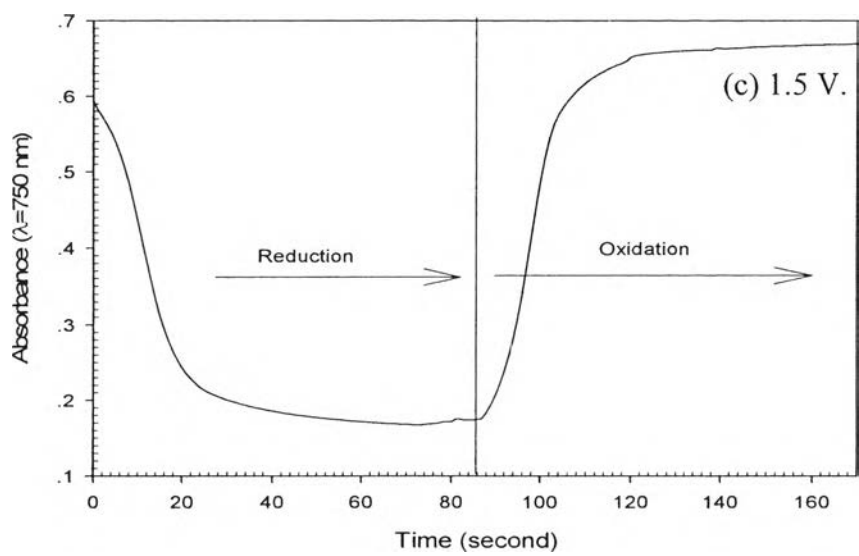
Figure E5 The absorption spectra of PDMA coated ITO plastic via hydrochloric acid for 10 minutes at (a) oxidized state applied potential of +1.0 V and (b) reduced state applied potential of -1.0 V.

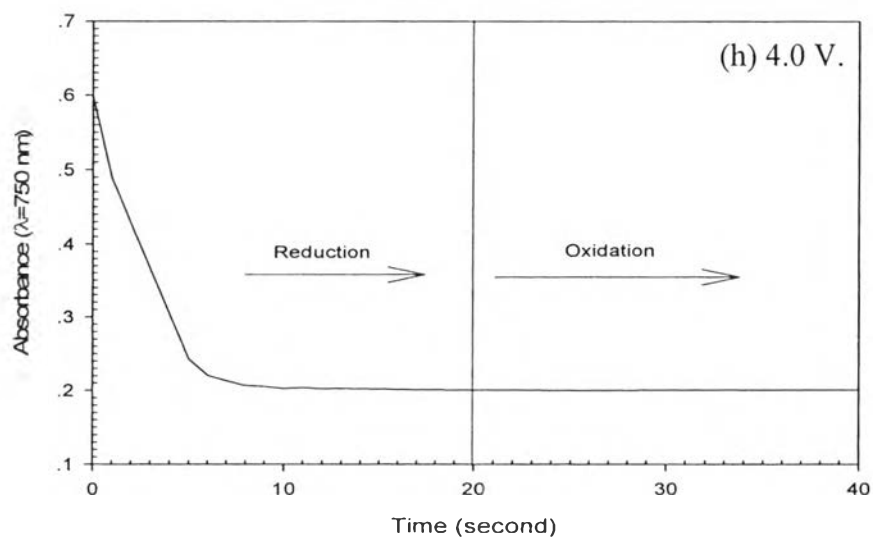
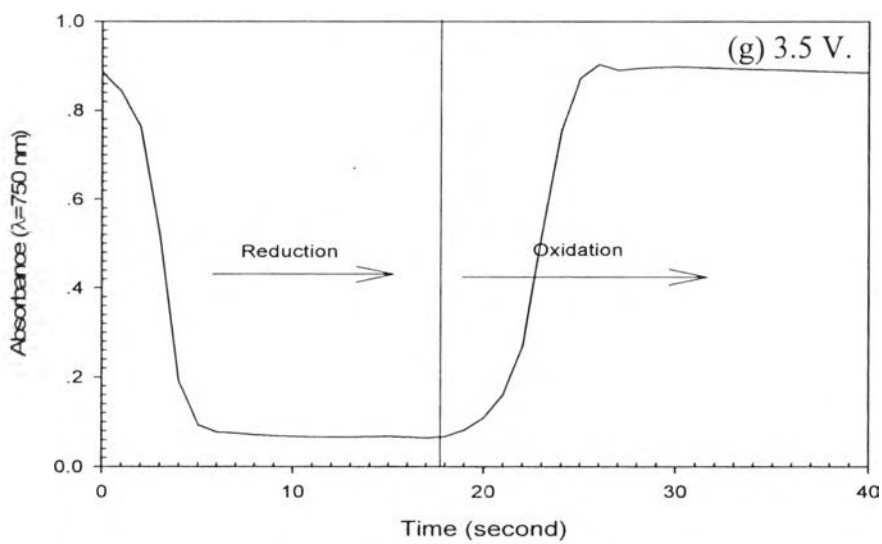
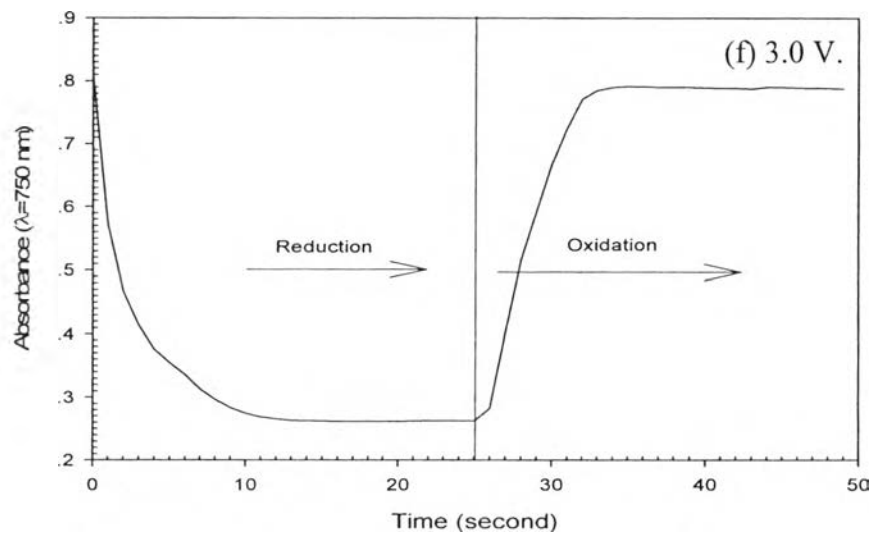
The study of the kinetic coloration of PDMA coated ITO electrode was carried out via the kinetic mode of UV–visible spectroelectrometer. The wavelength 750 nm representing the oxidized state was used to study the response time and optical density between the redox states. The measurement was set to collect 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 response times of PDMA synthesized at 3 minutes in oxalic acid at various potentials are shown in Figures E6 (a) to (j) and the data are tabulated in Table E1.







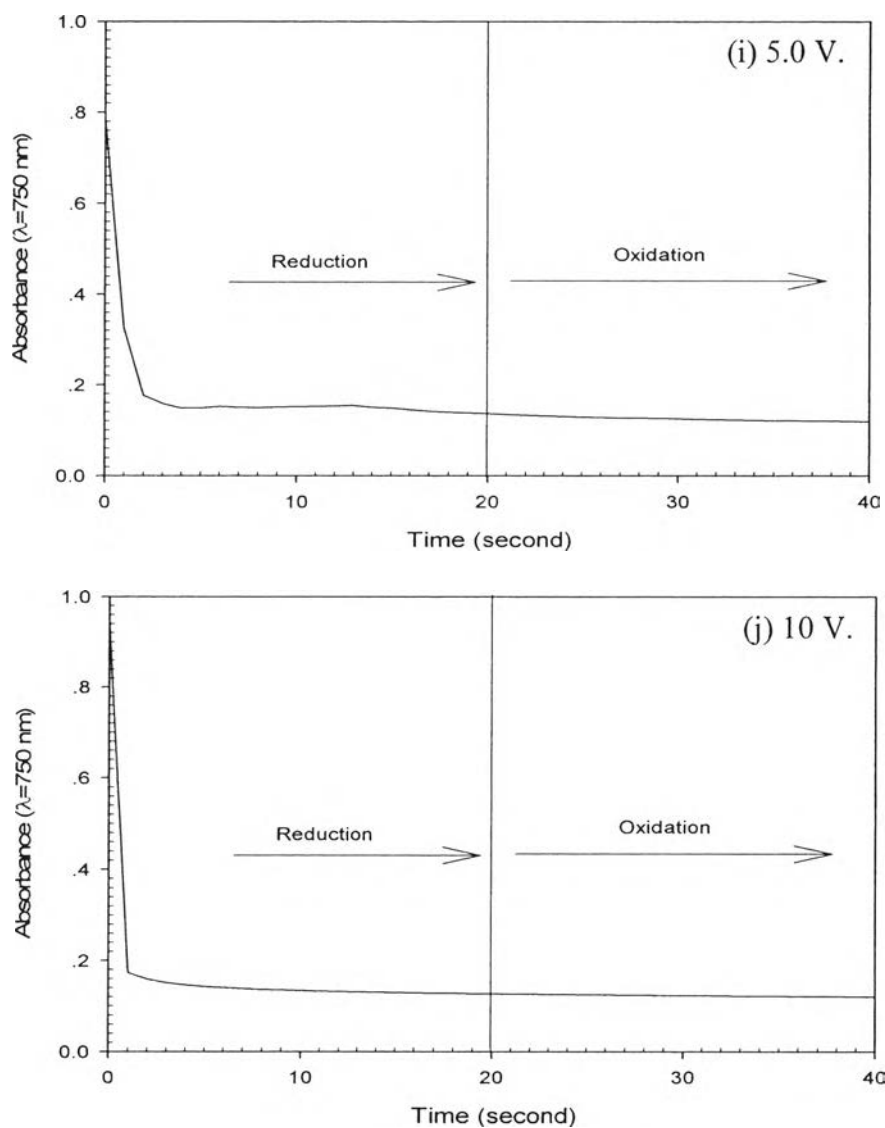


Figure E6 The response times of PDMA synthesized in oxalic acid at 3 minutes with testing potentials (a) 0.5, (b) 1.0, (c) 1.5, (d) 2.0, (e) 2.5, (f) 3.0, (g) 3.5, (h) 4.0, (i) 5.0, and (j) 10 V via UV-Visible spectrophotometer.

The reduction time was taken to be time required to reach the steady state value of the reduction state on the absorbance axis plus 5 % of the absorbance value. The result of the calculation leads to the reduction time, as shown in Figure E7.

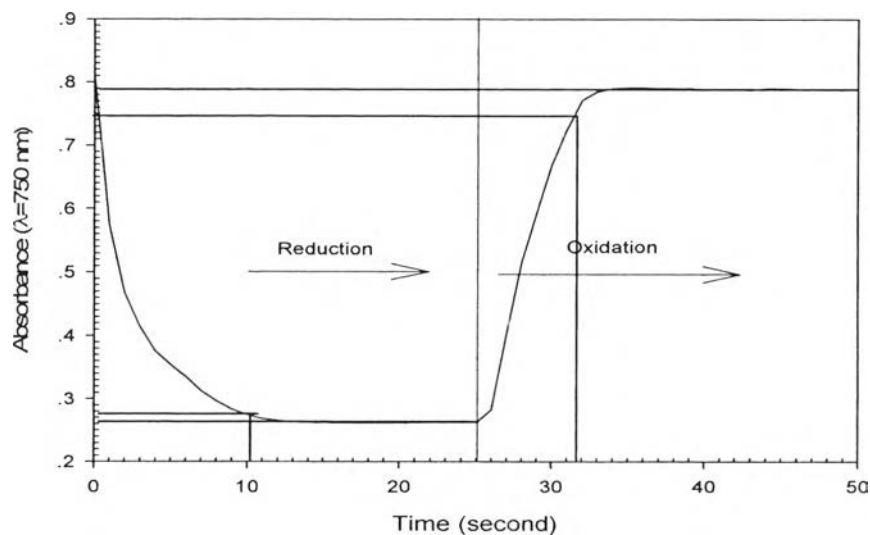


Figure E7 The determination of the reduction and oxidation times of PDMA synthesized in oxalic acid at 3 minutes with testing potential 3.0 V.

$$\begin{aligned}
 \text{The absorbance at steady reduction state} &= 0.26 \\
 5\% \text{ plus to } 0.26 &= 0.26 \times 1.05 \\
 &= 0.273
 \end{aligned}$$

The absorbance at 0.237 is the absorbance value at the time of 10 seconds representing the reduction time.

$$\begin{aligned}
 \text{The absorbance at steady oxidation state} &= 0.785 \\
 5\% \text{ deduct from } 0.785 &= 0.785 \times 0.95 \\
 &= 0.7457
 \end{aligned}$$

The absorbance at 0.7457 is the absorbance value at the time of 31.5 seconds when the oxidation starts at 25th second, so the oxidation time is 6.5 seconds. The total response time was determined by the summation of the reduction and the oxidation times, in this case, the total response time is 16.5 seconds.

Table E1 The reduction, oxidation and total response times of PDMA synthesized in oxalic acid for 3 minutes at various testing potentials

Testing potential (V)	Reduction time (sec)	Oxidation time (sec)	Total response time (sec)
0.5	-	-	-
1.0	110	170	180
1.5	47	28.7	75.7
2.0	24	12	36
2.5	22	7.5	29.5
3.0	10	6.5	16.5
3.5	6.8	6.0	12.8
4.0	6.0	-	-
5.0	3.3	-	-
10	2.5	-	-

At the potential 1.5 V, the kinetic experiment was repeated 3 times in order to determine the average value and the standard deviation (SD) value of the response time of PDMA/ITO synthesized for 3 minutes in oxalic acid, and the results are shown in Figure E8 and the response times are tabulated in Table E2.

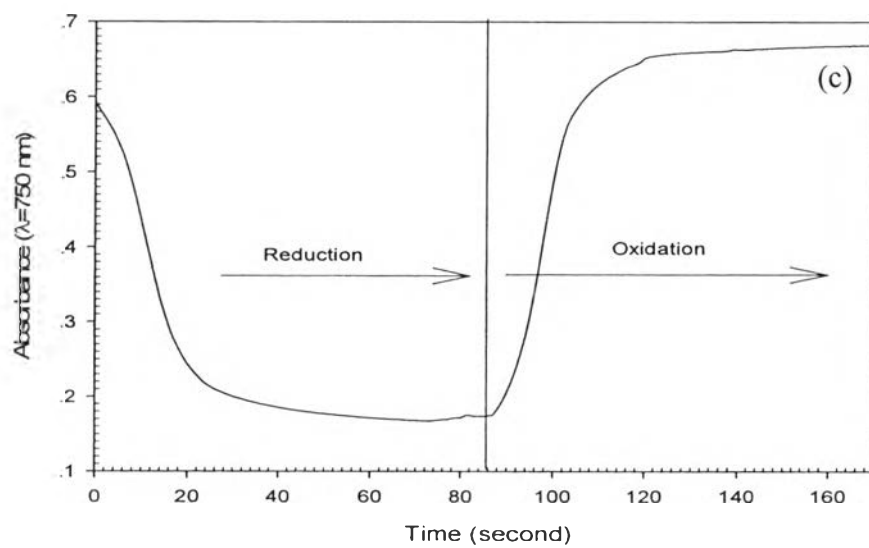
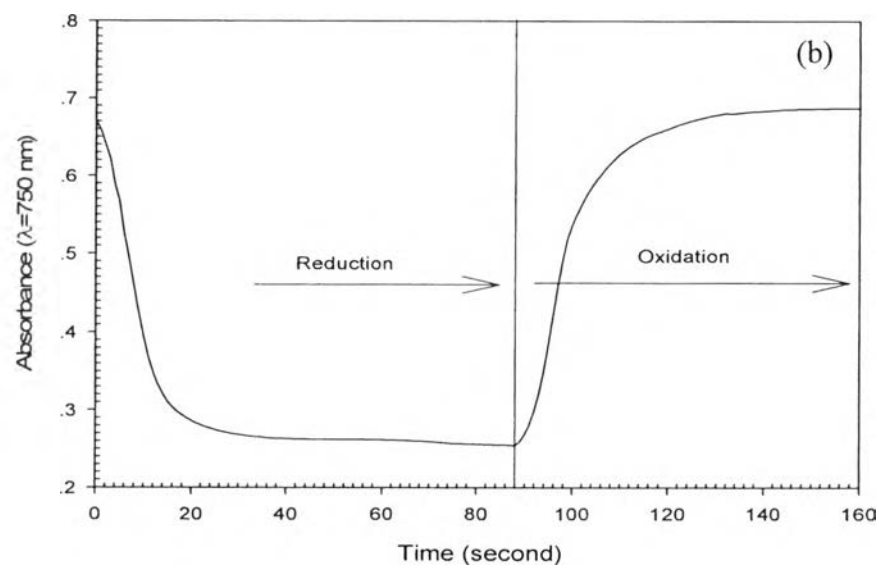
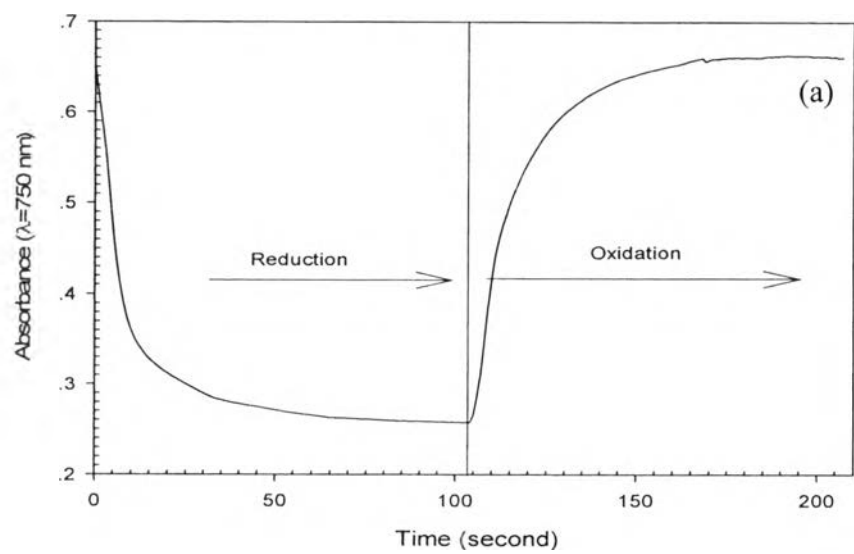
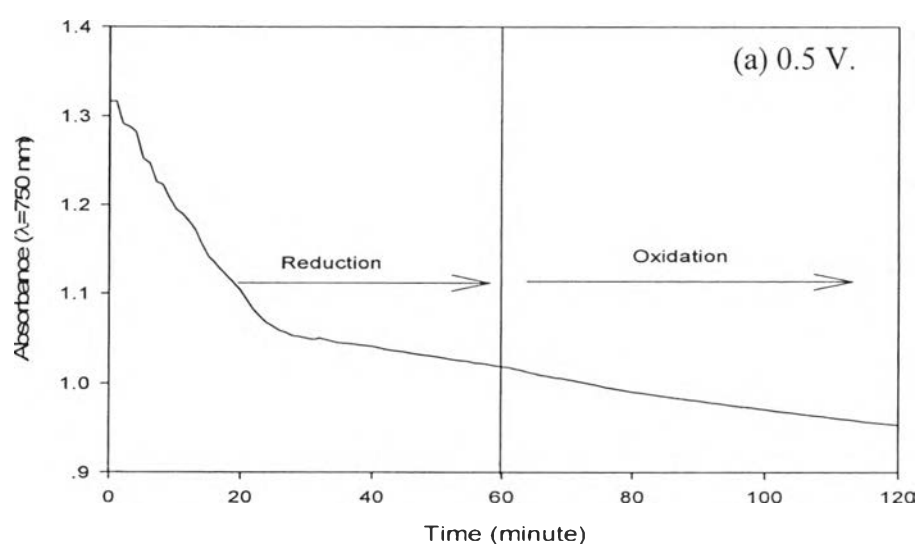


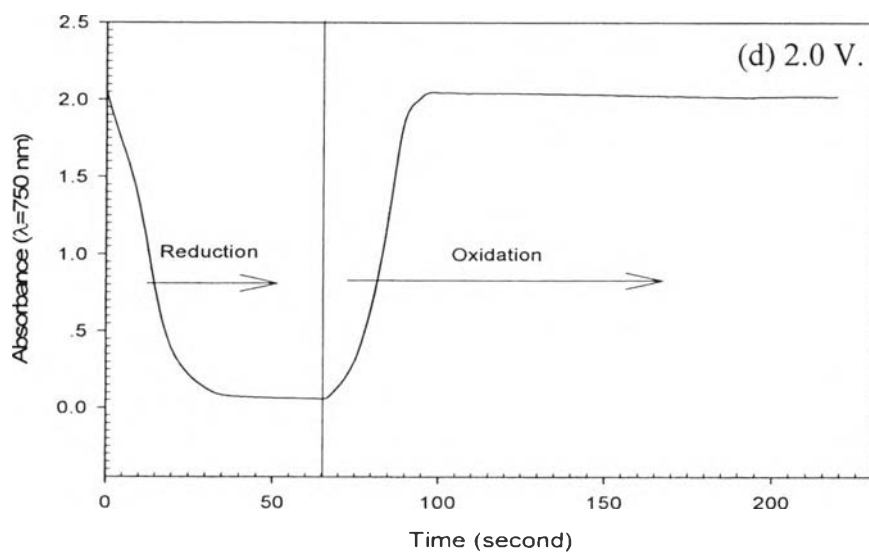
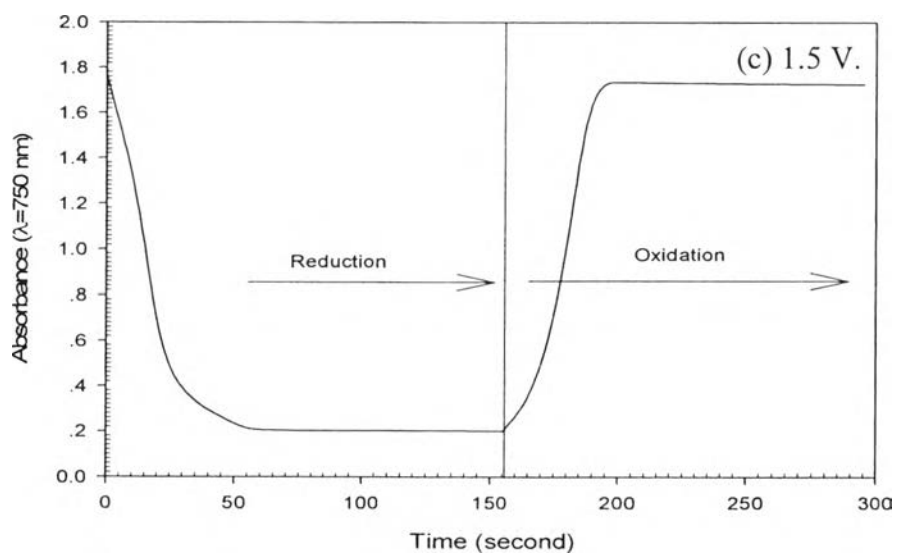
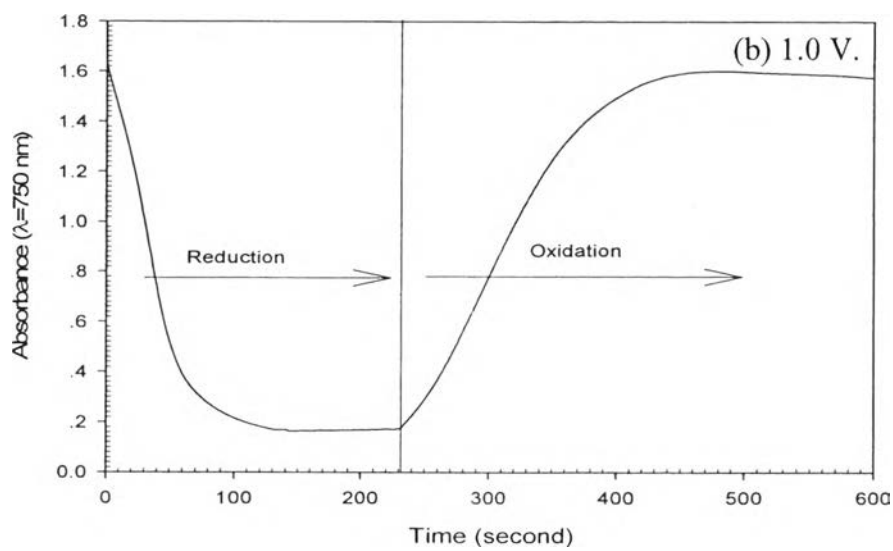
Figure E8 The response time of PDMA synthesized in oxalic acid at 3 minutes with 3 times repeating experiments (a) to (c) at testing potential 1.5 V.

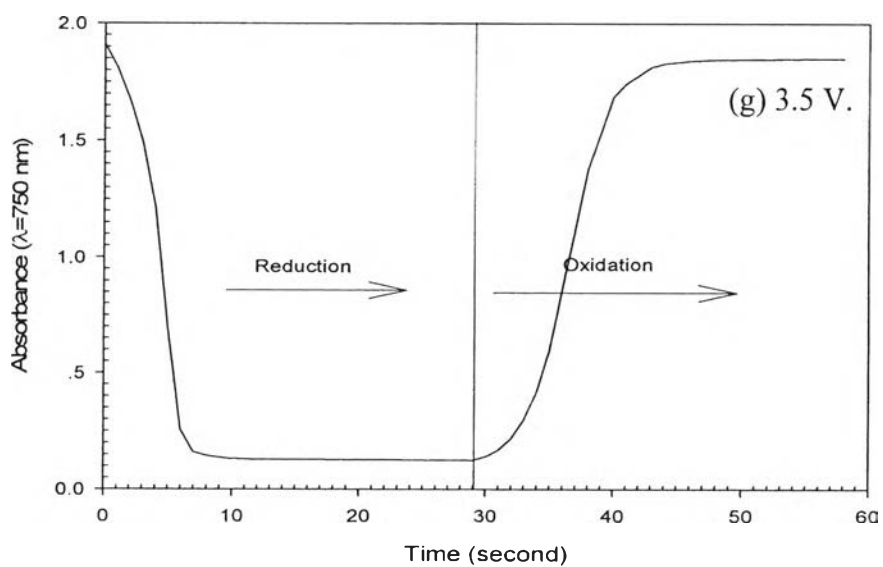
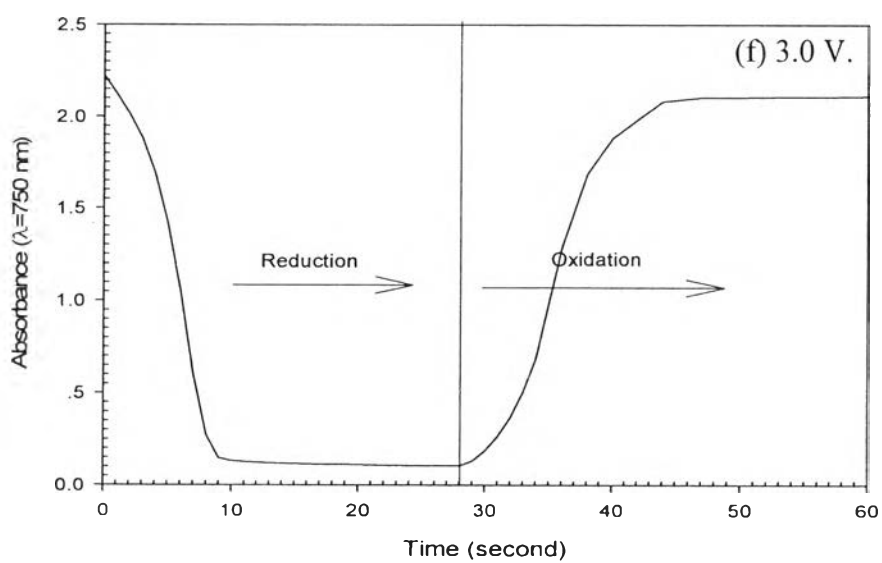
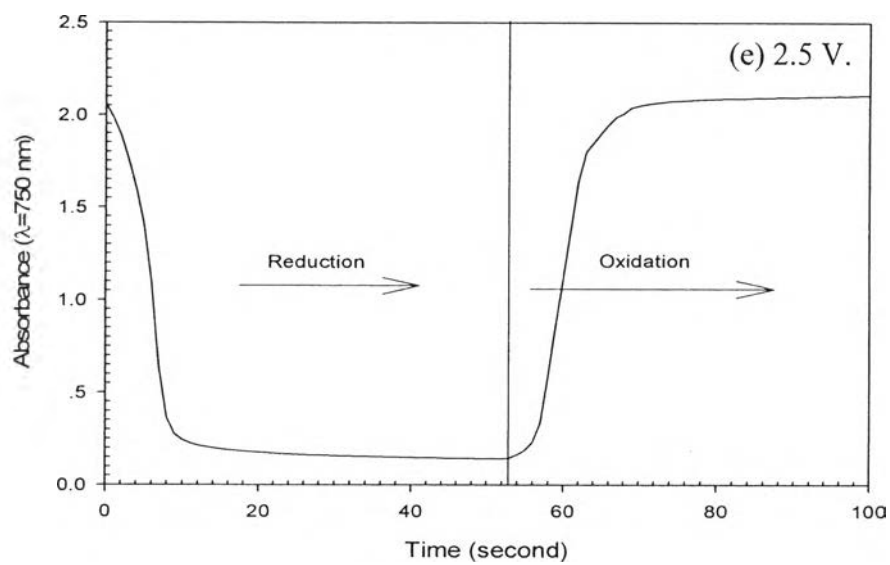
Table E2 The response time of PDMA/ITO synthesized in oxalic acid for 3 minutes with testing potential 1.5 V from the repeating experiments

Testing times at 1.5 V of PDMA films	Response time (sec)		
	reduction	oxidation	total
1 st	50	32	82
2 nd	44	26	70
3 rd	47	28	75
Average	47 ± 3.0	28.7 ± 3.06	75.7 ± 6.03

The absorption curves of PDMA/ITO synthesized for 6 minutes in oxalic acid at 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 5.0, and 10 V. are shown in Figures E9 (a) to (j), respectively, and the data are tabulated in Table E3.







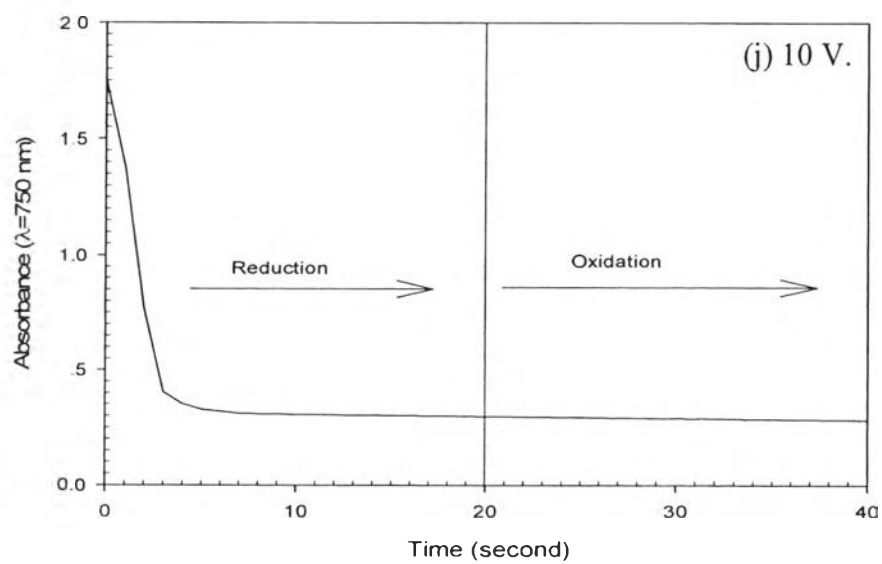
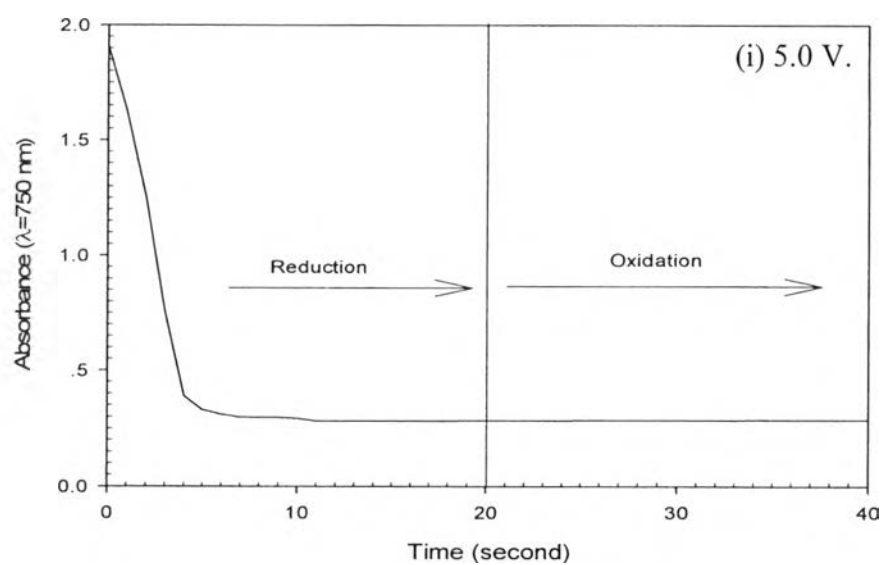
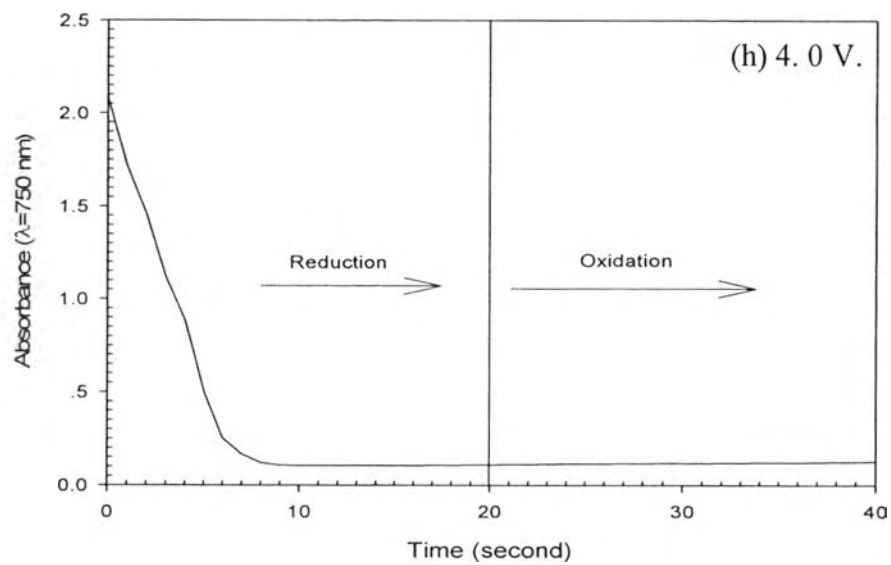


Figure E9 The response time of PDMA synthesized in oxalic acid at 6 minutes with testing potentials (a) 0.5, (b) 1.0, (c) 1.5, (d) 2.0, (e) 2.5, (f) 3.0, (g) 3.5, (h) 4.0, (i) 5.0, and (j) 10 V via UV-Visible spectrophotometer.

Table E3 The reduction, oxidation and total response time of PDMA synthesized in oxalic acid for 6 minutes at various testing potentials

Testing potential (V)	Reduction time (sec)	Oxidation time (sec)	Total response time (sec)
0.5	-	-	-
1.0	110	180	290
1.5	52.3	34.7	87
2.0	34	27	61
2.5	15	13	28
3.0	10	14	24
3.5	9	12	21
4.0	7.8	-	-
5.0	7	-	-
10	6	-	-

At the potential of 1.5 V., the kinetic experiment was repeated 3 times in order to determine the average value and the standard deviation (SD) value of the response time of PDMA/ITO synthesized for 6 minutes in oxalic acid. The results are shown in Figure E10 and the response times are tabulated in Table E4.

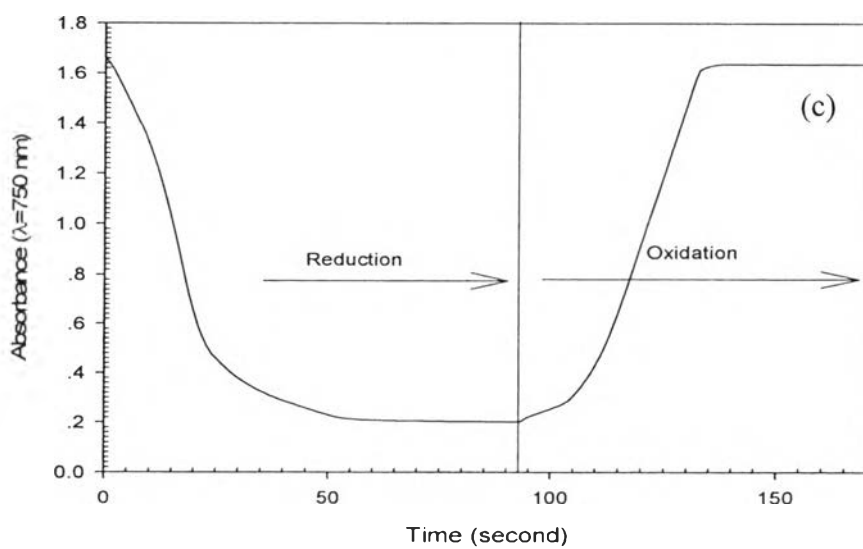
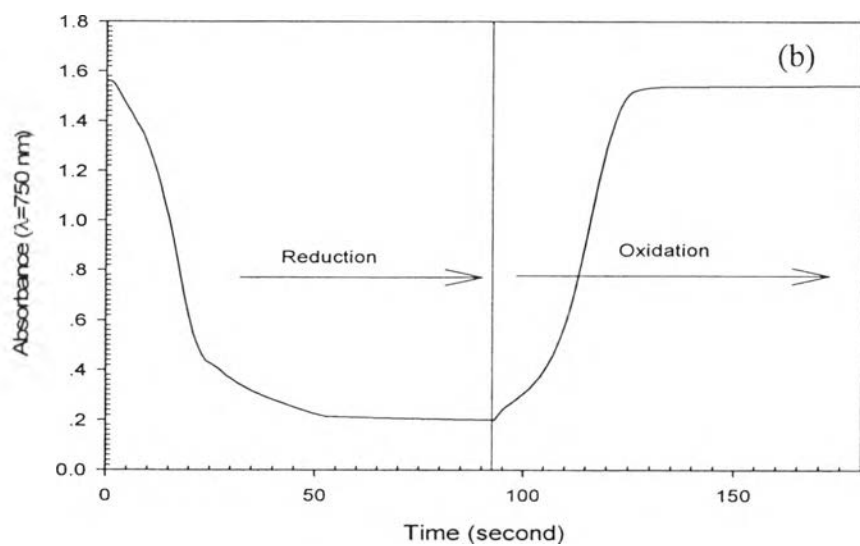
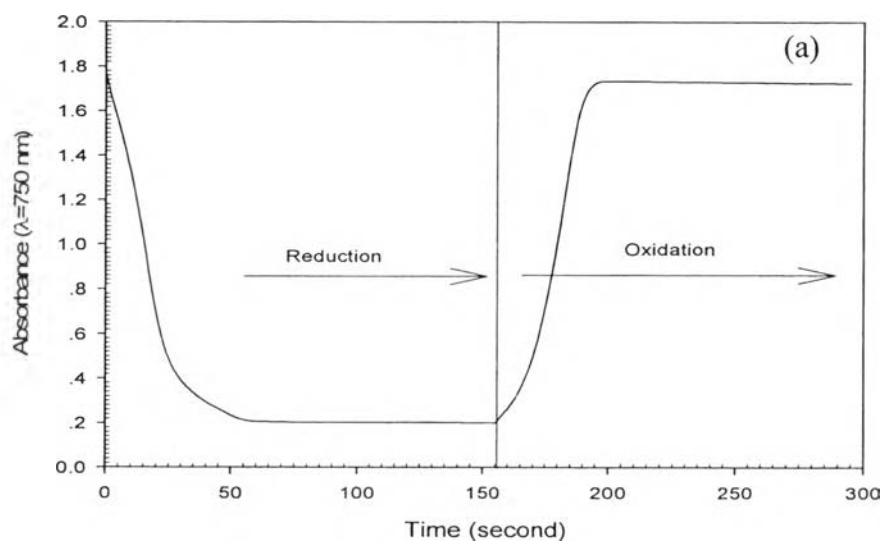
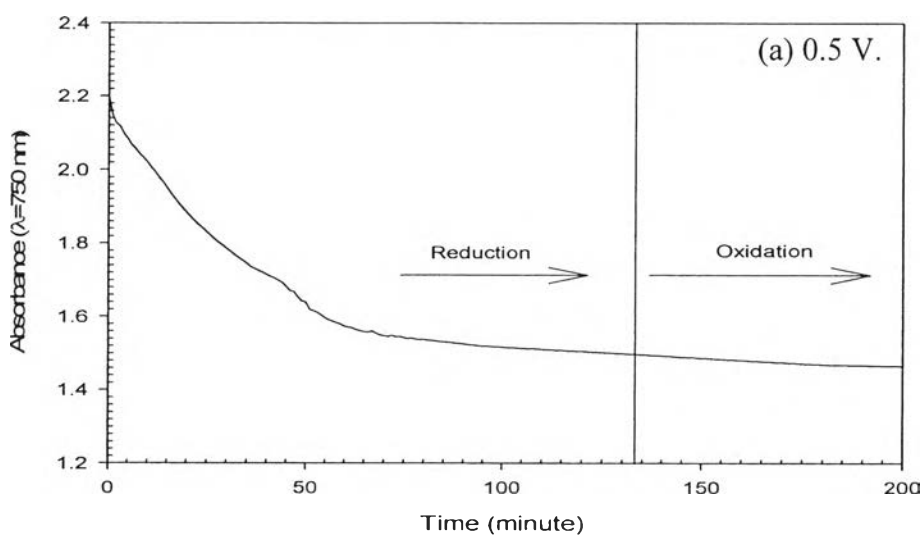


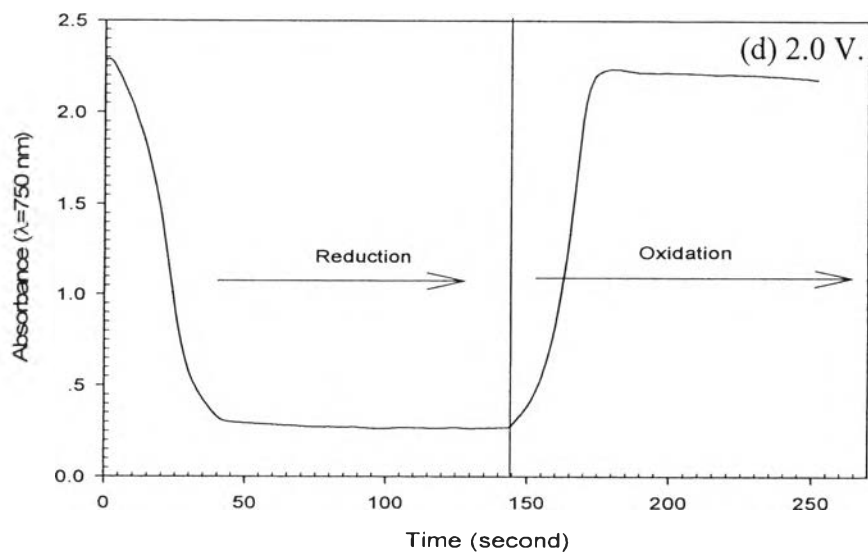
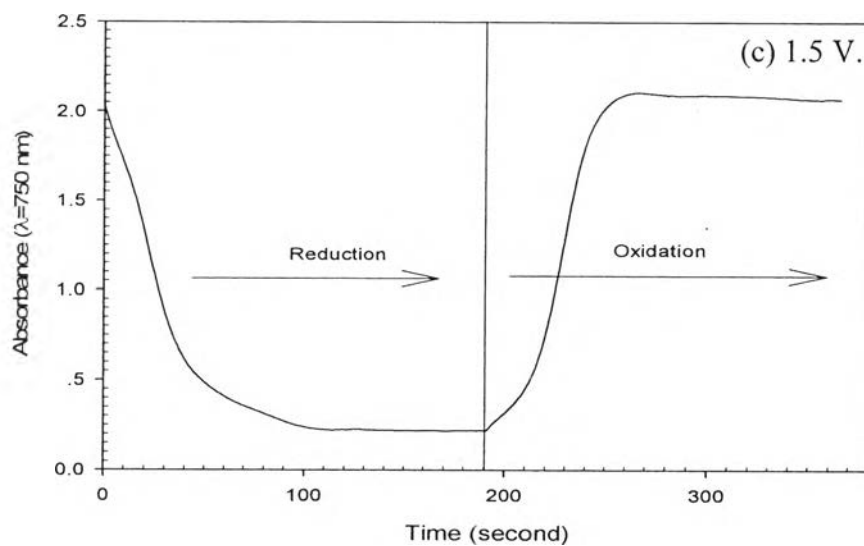
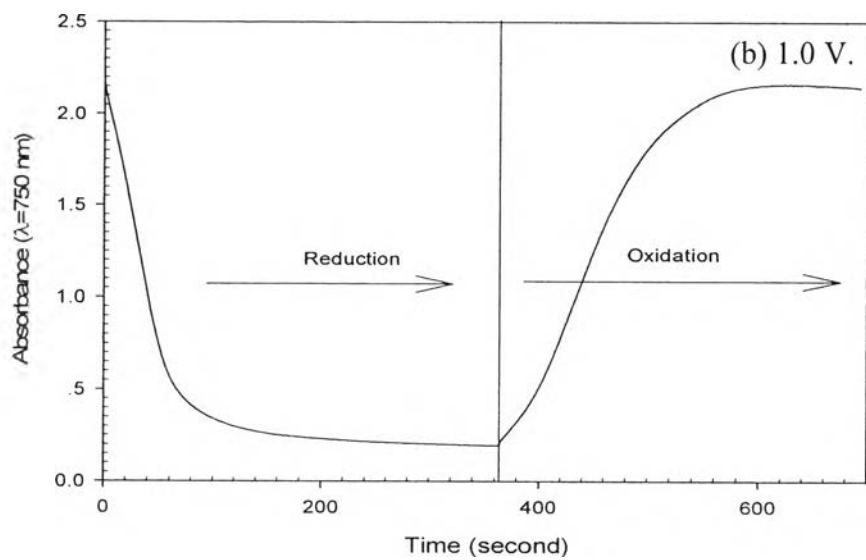
Figure E10 The response time of PDMA synthesized in oxalic acid at 6 minutes with 3 times repeating experiments (a) to (c) at the testing potential 1.5 V.

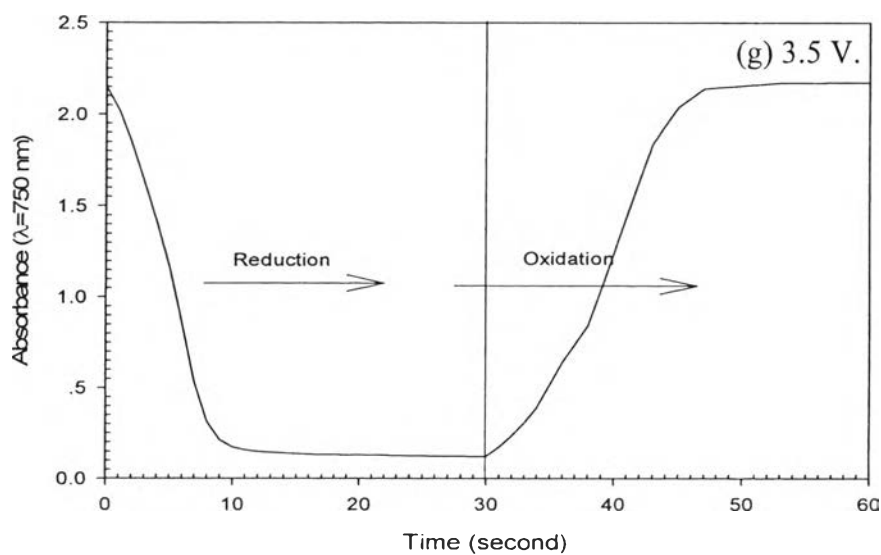
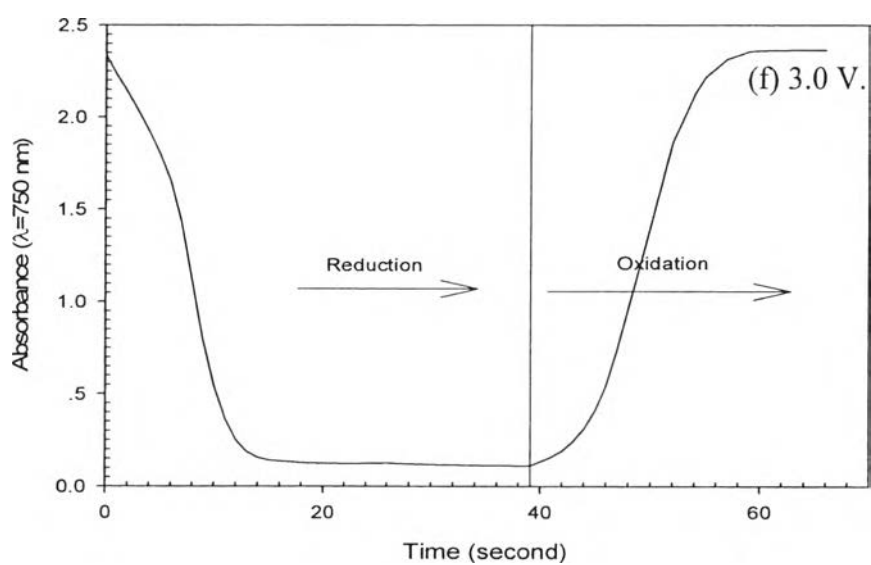
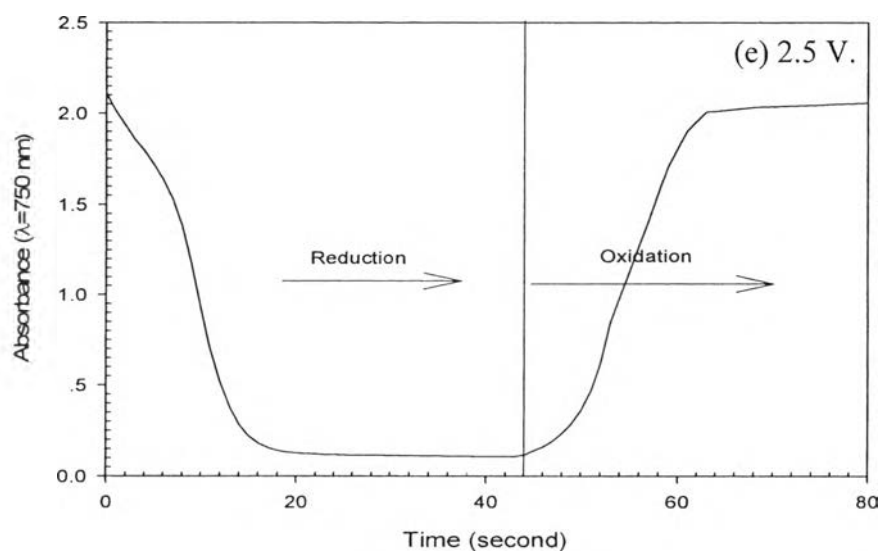
Table E4 The response time of PDMA/ITO synthesized in oxalic acid for 6 minutes with testing potential 1.5 V in the repeating experiments

Testing times at 1.5 V of PDMA films	Response time (sec)		
	reduction	oxidation	total
1 st	53	35	88
2 nd	50	30	80
3 rd	54	39	93
Average	52.3 ± 2.08	34.7 ± 4.51	87 ± 4.5

The absorption curves of PDMA/ITO synthesized for 10 minutes in oxalic acid at 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 5.0, and 10 V are shown in Figures E11 (a) to (j), respectively. The response data are tabulated in Table E5.







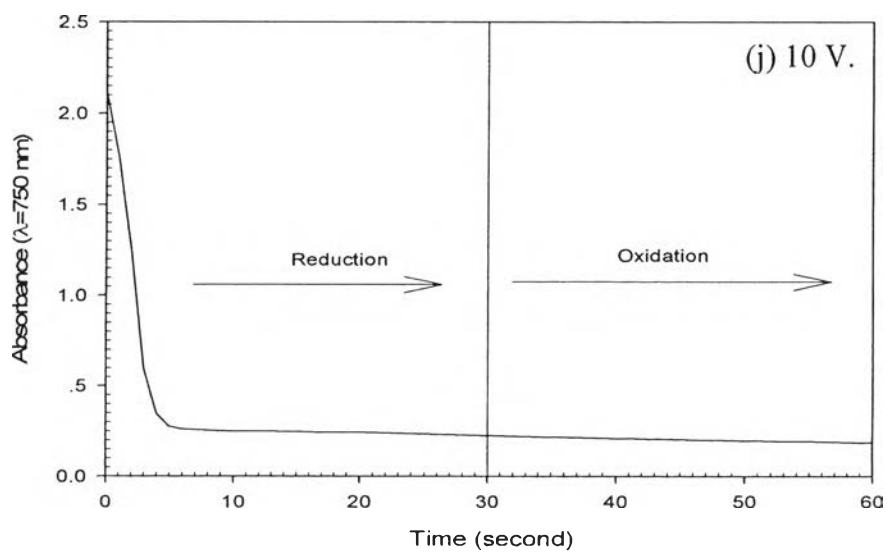
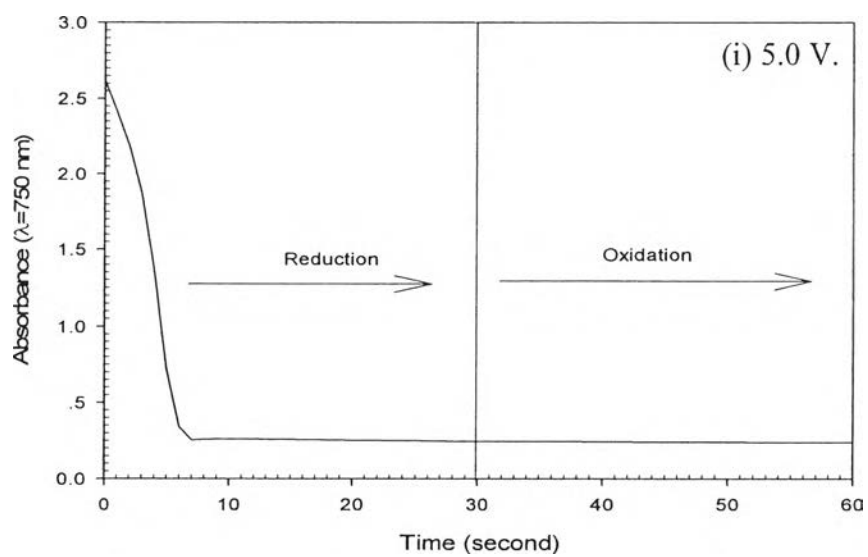
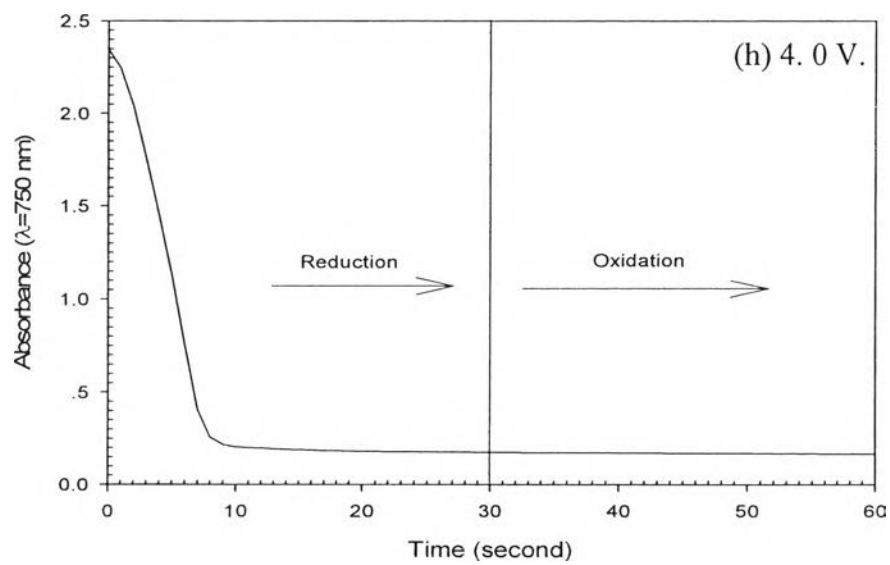


Figure E11 The response times of PDMA synthesized in oxalic acid at 10 minutes with testing potentials (a) 0.5, (b) 1.0, (c) 1.5, (d) 2.0, (e) 2.5, (f) 3.0, (g) 3.5, (h) 4.0, (i) 5.0, and (j) 10 V via UV-Visible spectrophotometer.

Table E5 The reduction, oxidation and total response time of PDMA synthesized in oxalic acid for 10 minutes at various testing potentials

Testing potential (V)	Reduction time (sec)	Oxidation time (sec)	Total response time (sec)
0.5	-	-	-
1.0	210	175	385
1.5	100	62.7	162.7
2.0	70	28	98
2.5	19.5	17	36.5
3.0	17	16	33
3.5	11.5	14	25.5
4.0	9	-	-
5.0	6.5	-	-
10	5	-	-

At the potential 1.5 V., the kinetic experiment was repeated 3 times in order to determine the average and the standard deviation (SD) values of the response time of PDMA/ITO synthesized for 10 minutes in oxalic acid, and the results are shown in Figure E12 and the response times are tabulated in Table E6.

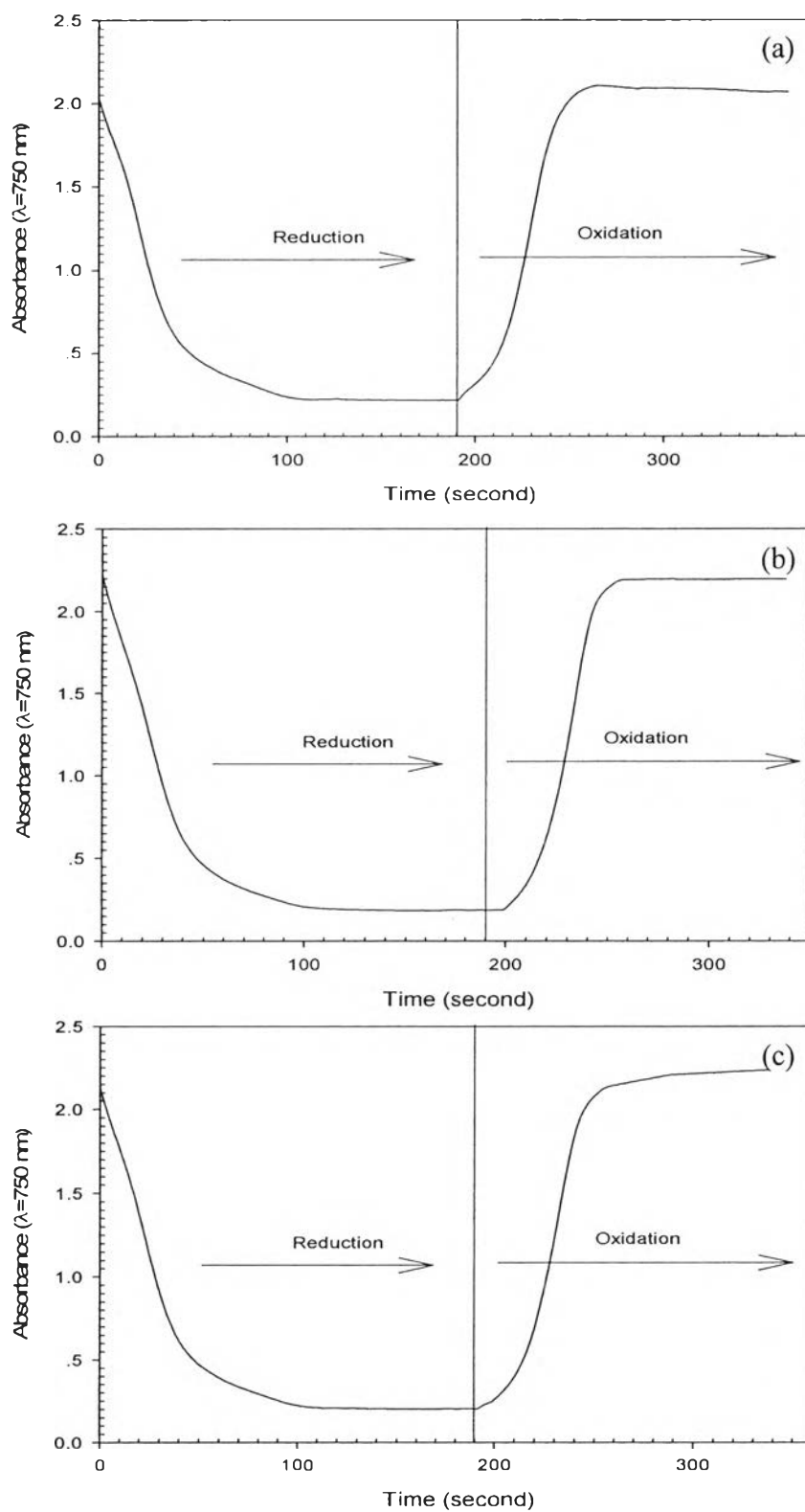


Figure E12 The response times of PDMA synthesized in oxalic acid at 10 minutes with 3 times repeating experiments (a) to (c) at testing potential 1.5 V.

Table E6 The response times of PDMA/ITO synthesized in oxalic acid for 10 minutes with testing potential 1.5 V. in the repeating experiments

Testing times at 1.5 V of PDMA films	Response time (sec)		
	reduction	oxidation	total
1 st	95	58	153
2 nd	100	62	162
3 rd	105	68	173
Average	100 ± 5	62.7 ± 5.03	162.7 ± 10.02

The effect of the testing potentials on the reduction, oxidation and response times of PDMA synthesized in oxalic acid at various dipping times is shown in Figures E13-E15.

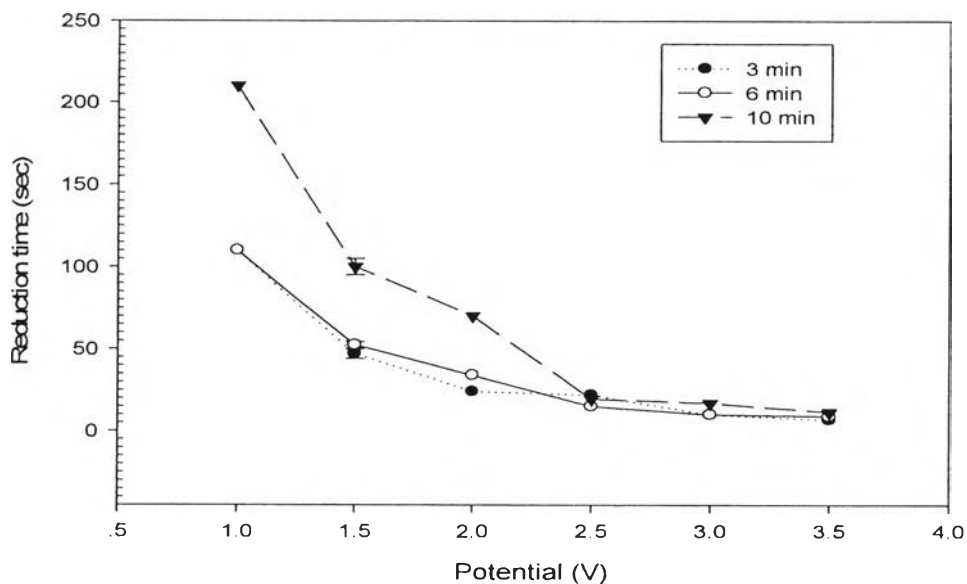


Figure E13 The effect of the testing potentials on the reduction time of PDMA synthesized in oxalic acid at various dipping times.

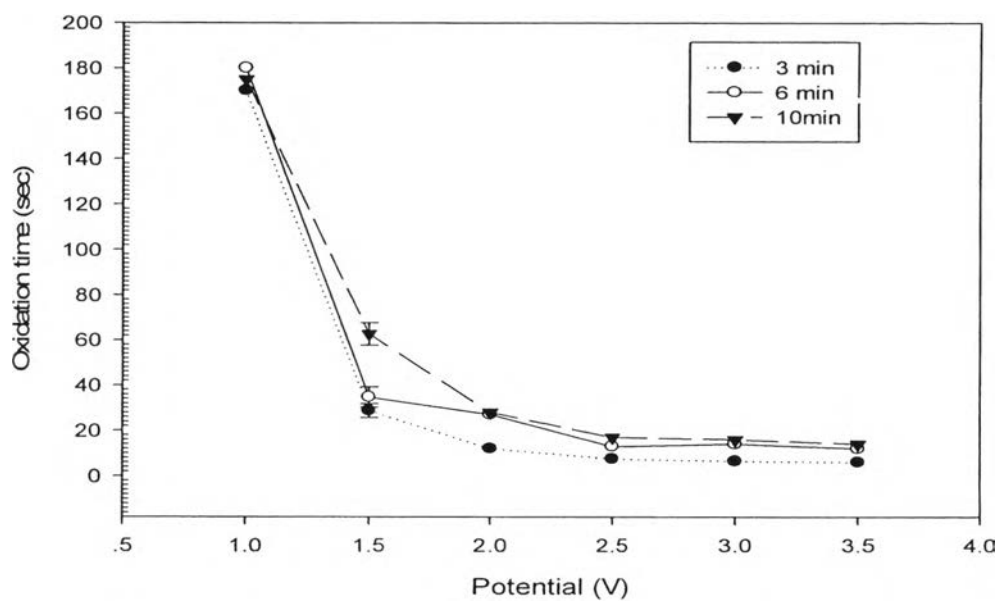


Figure E14 The effect of the testing potentials on the oxidation time of PDMA synthesized in oxalic acid at various dipping times.

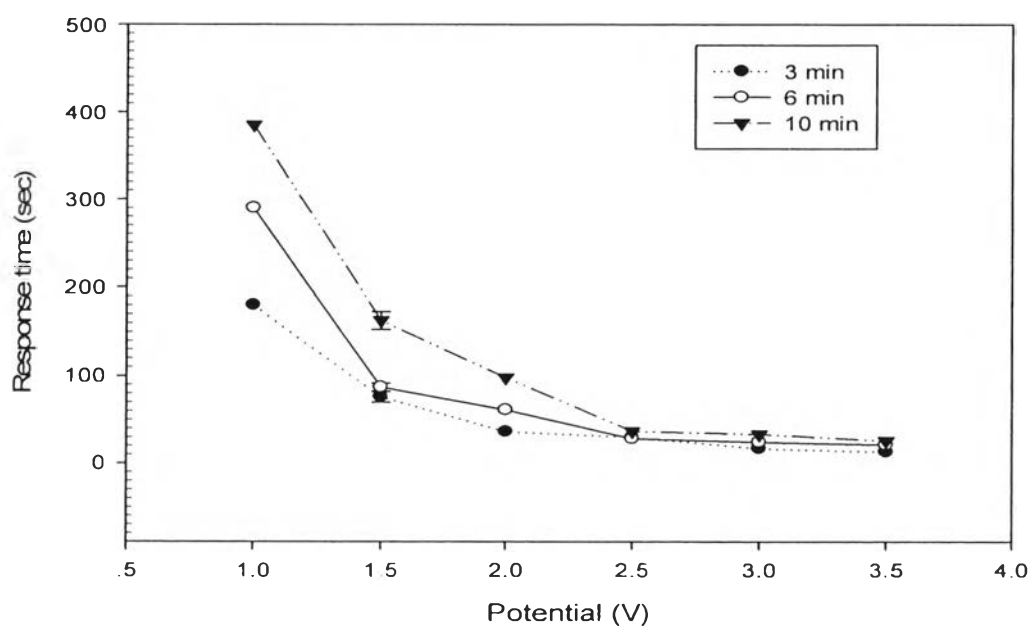
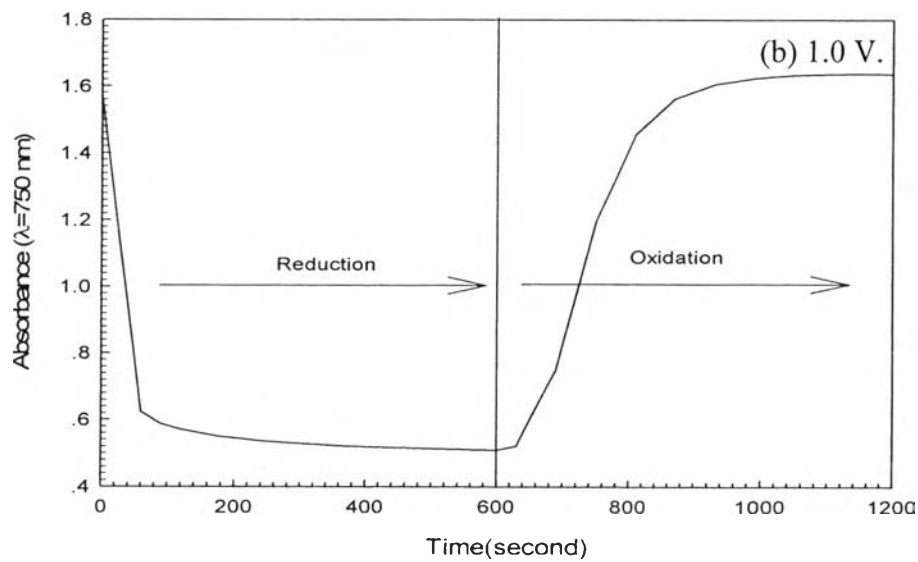
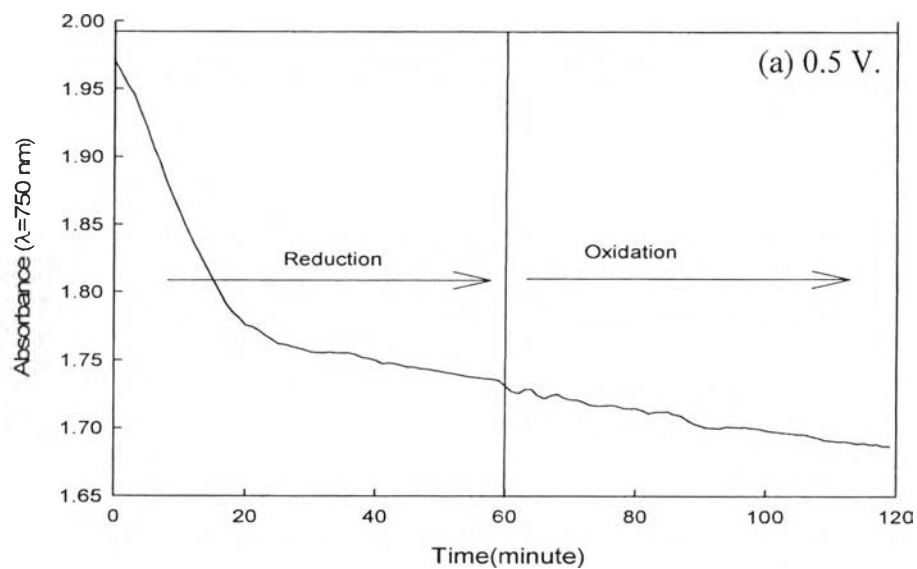
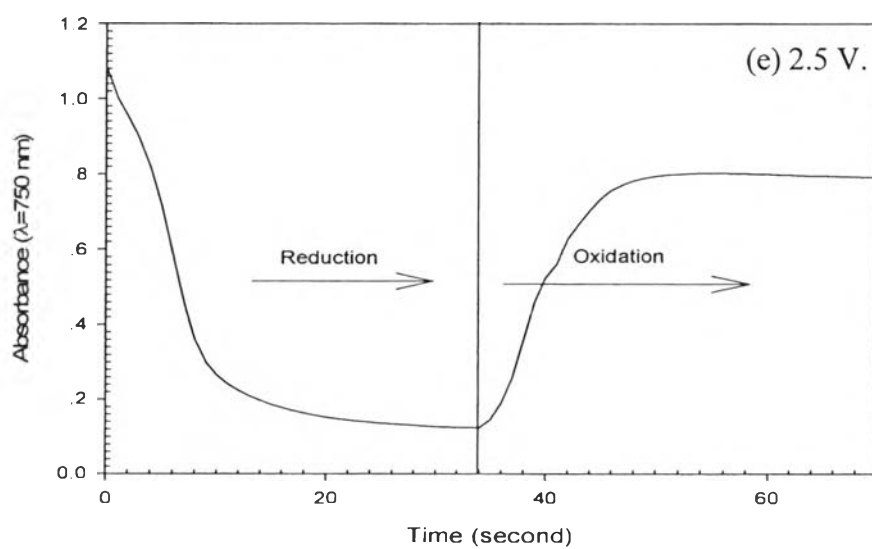
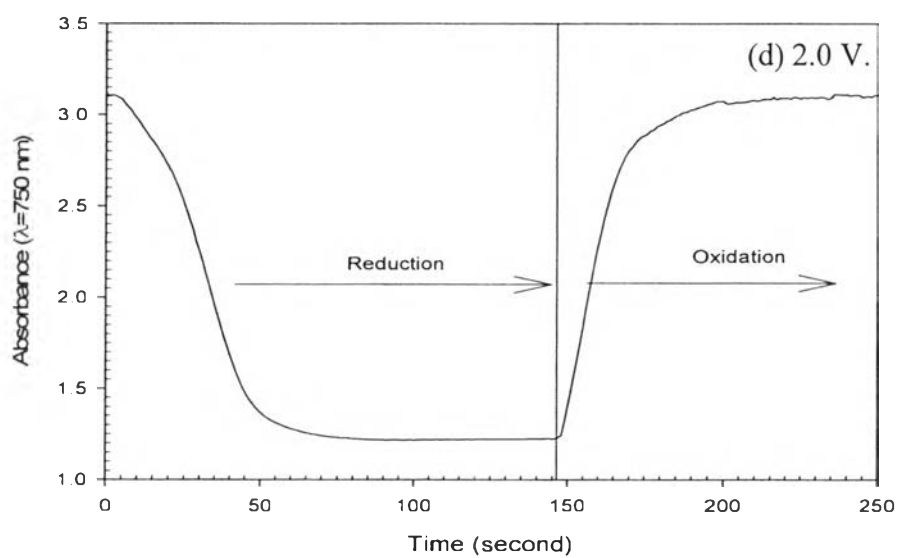
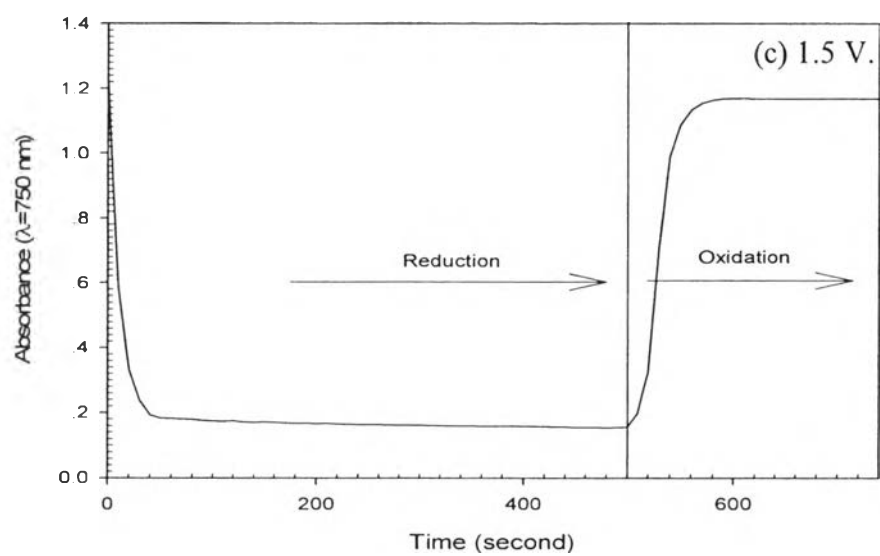
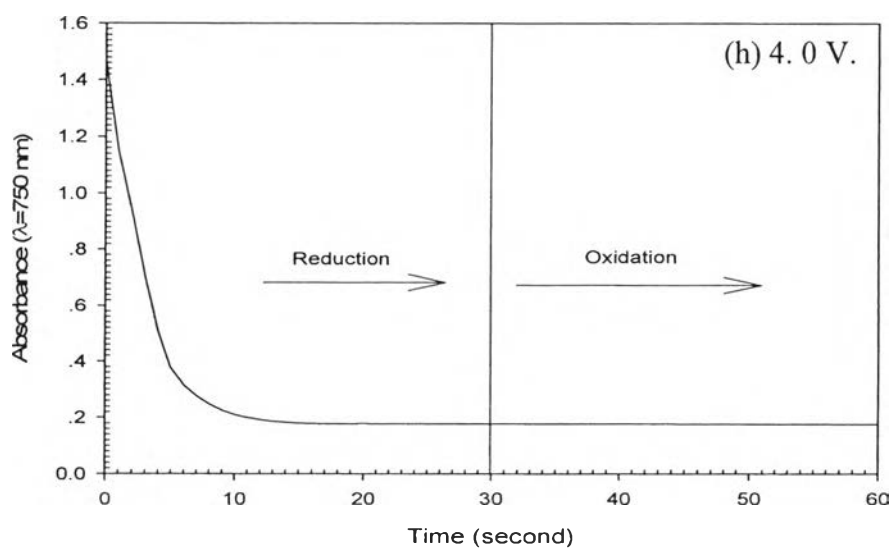
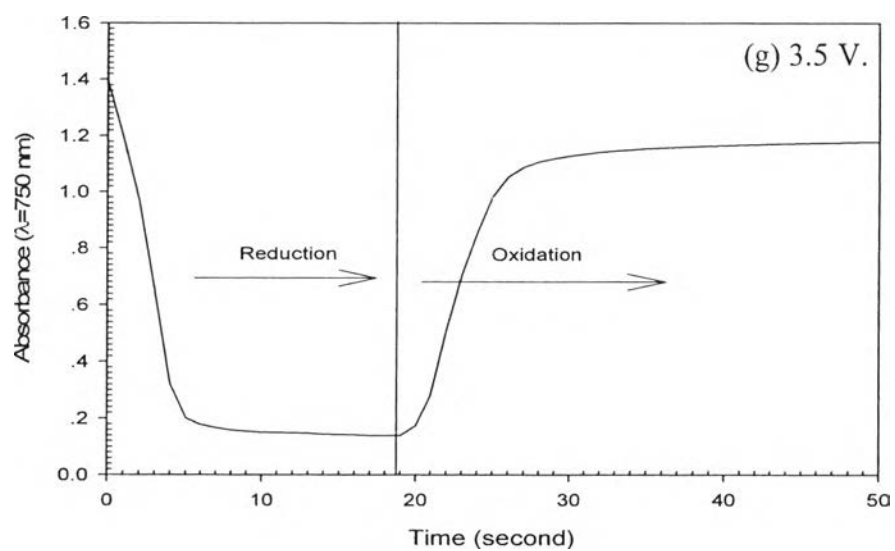
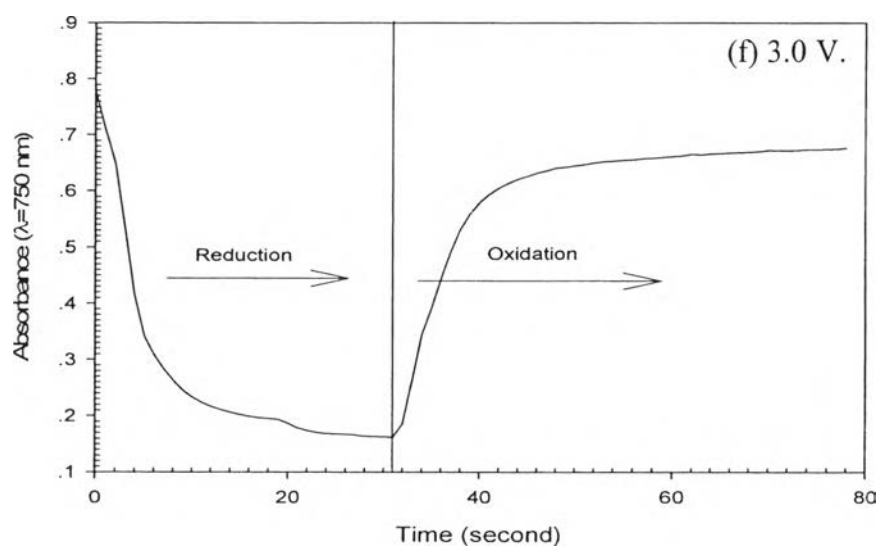


Figure E15 The effect of the testing potentials on the response time of PDMA synthesized in oxalic acid at various dipping times.

The response times of PDMA synthesized at 3 minutes in nitric acid at testing potentials 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 5.0 , and 10 V are shown in Figures E16 (a) to (j) and the data are tabulated in Table E7.







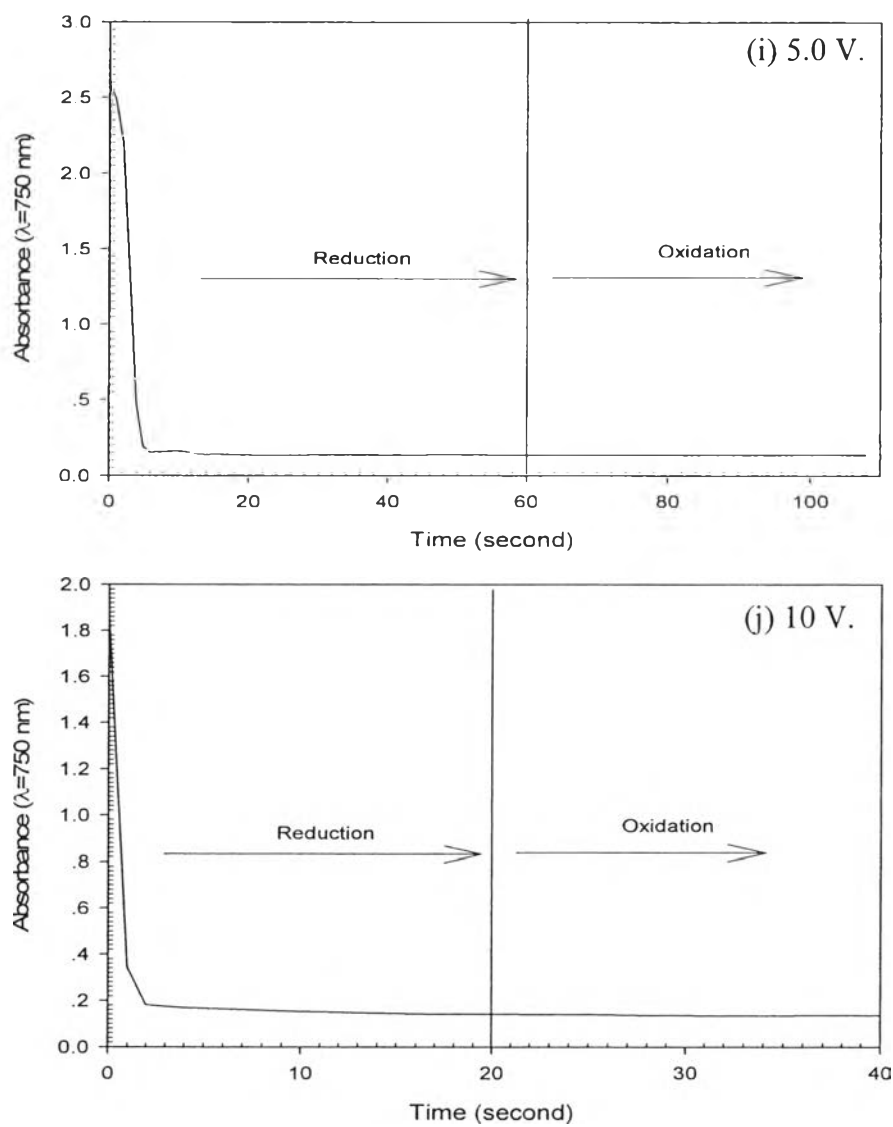
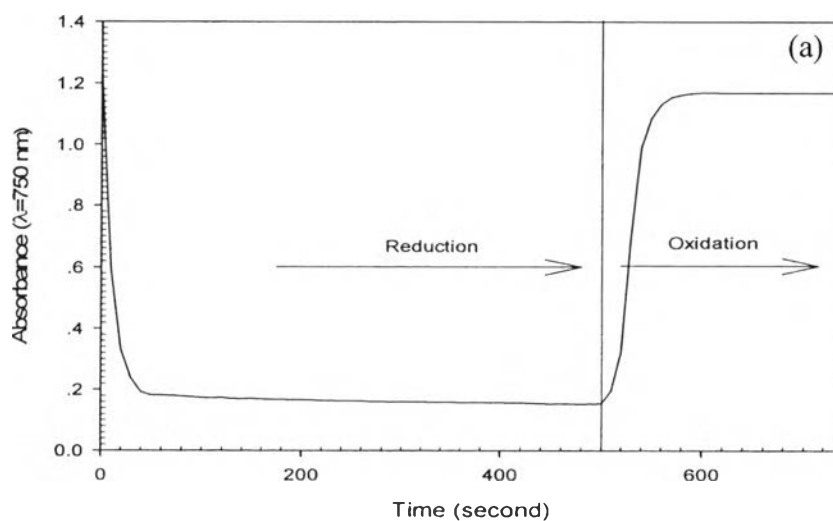


Figure E16 The response times of PDMA synthesized in nitric acid at 3 minutes with testing potentials (a) 0.5, (b) 1.0, (c) 1.5, (d) 2.0, (e) 2.5, (f) 3.0, (g) 3.5, (h) 4.0, (i) 5.0, and (j) 10 V via UV-Visible spectroelectrometer.

Table E7 The reduction, oxidation and response time of PDMA synthesized in nitric acid for 3 minutes at various testing potentials

Testing potential (V)	Reduction time (sec)	Oxidation time (sec)	Total response time (sec)
0.5	-	-	-
1.0	200	260	460
1.5	178.7	55.7	234.4
2.0	70	39	109
2.5	21	14	35
3.0	20	13	33
3.5	9.5	10.5	20
4.0	9	-	-
5.0	6	-	-
10	4.5	-	-

At the potential 1.5 V., the kinetic experiment was repeated 3 times in order to determine the average and the standard deviation (SD) values of the response time of PDMA/ITO synthesized for 3 minutes in nitric acid, and the results are shown in Figure E17 and the response times are tabulated in Table E8.



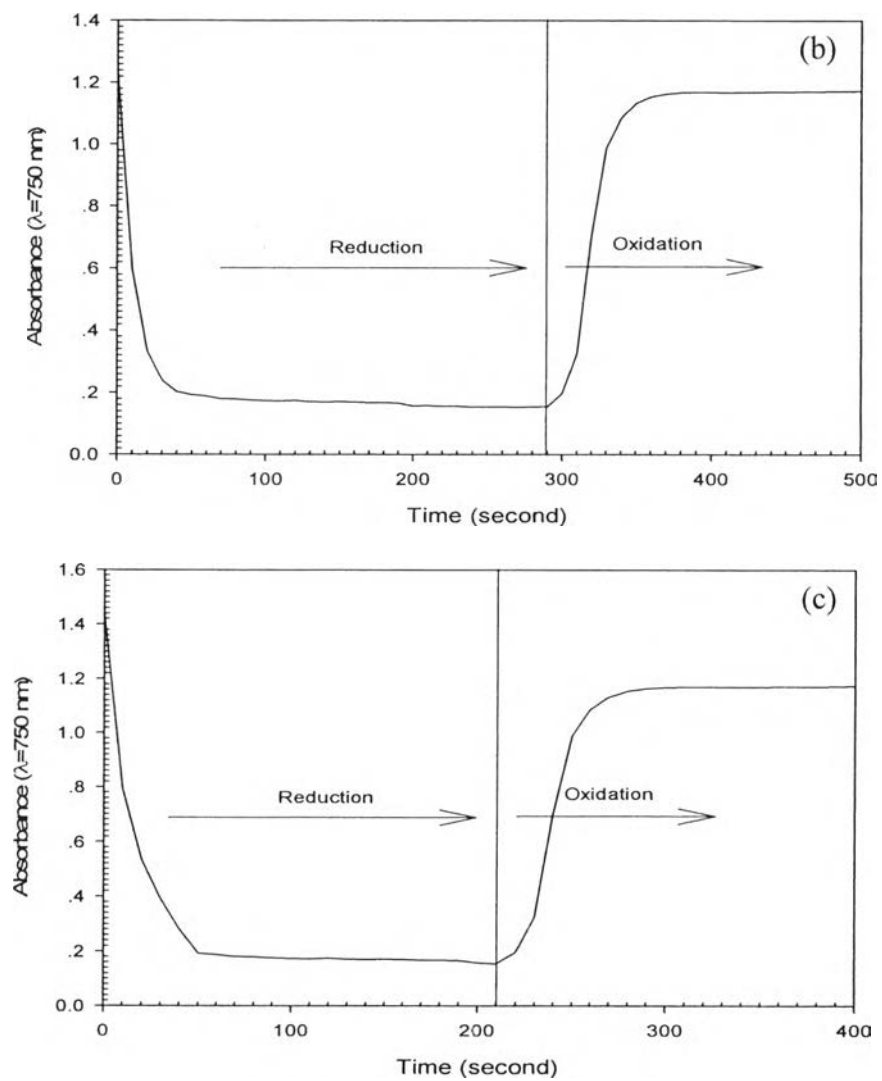
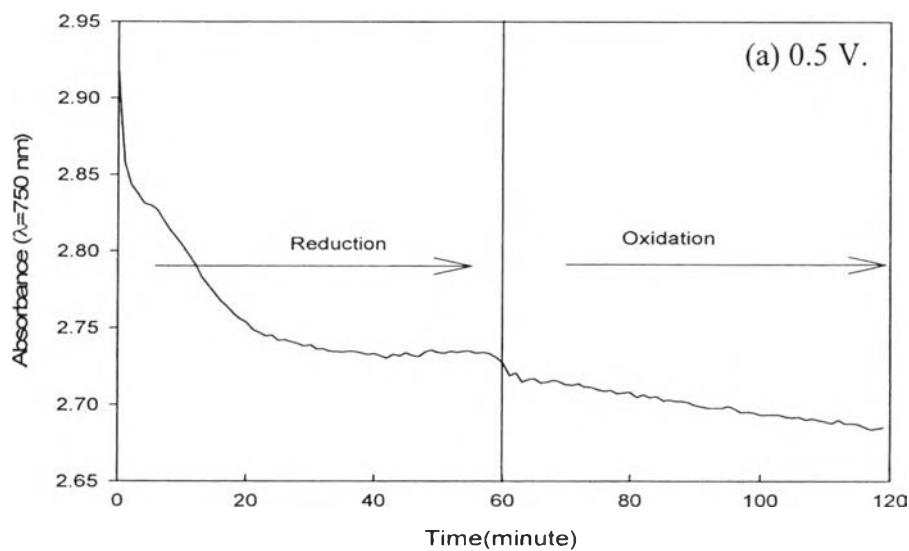


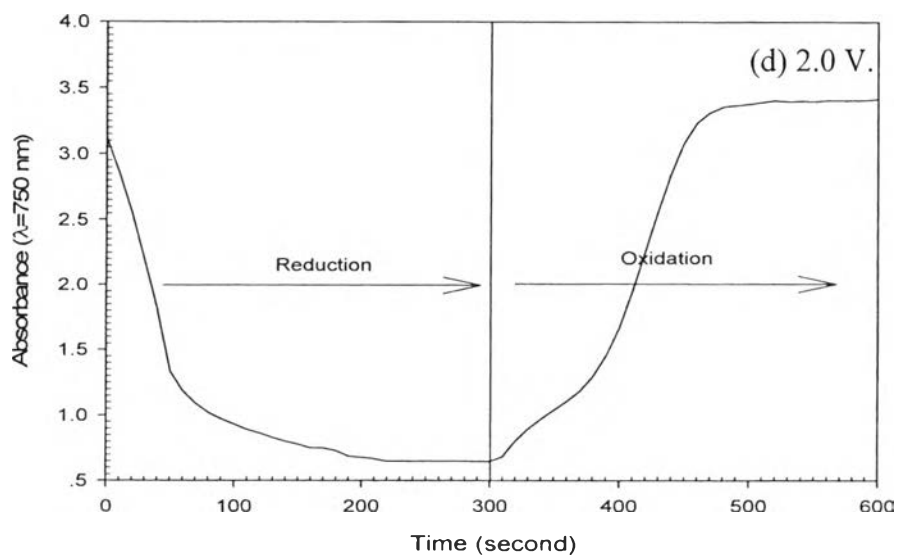
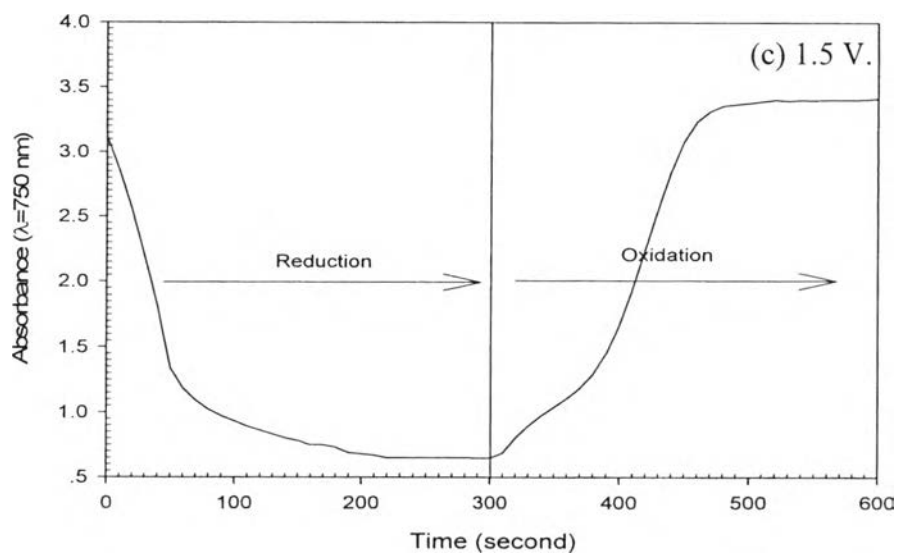
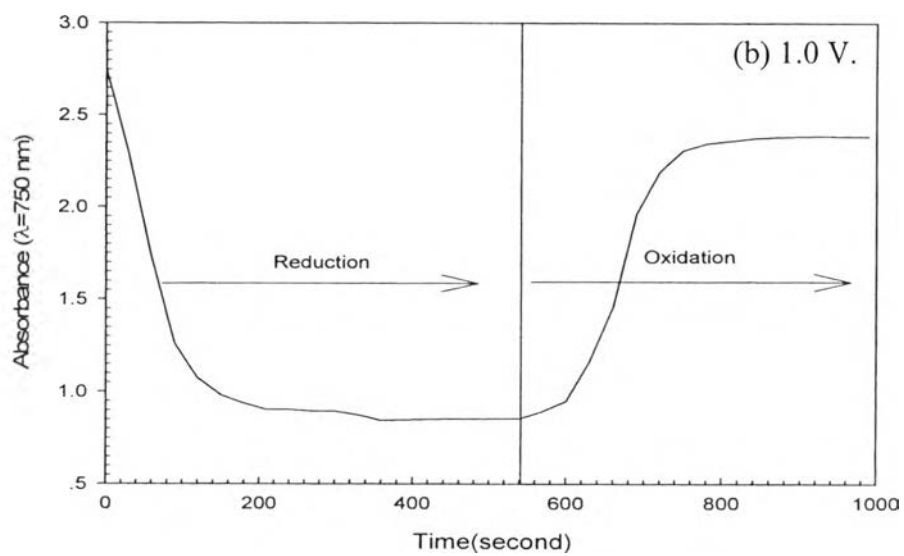
Figure E17 The response times of PDMA synthesized in nitric acid at 3 minutes with 3 times repeating experiments (a) to (c) at testing potential 1.5 V.

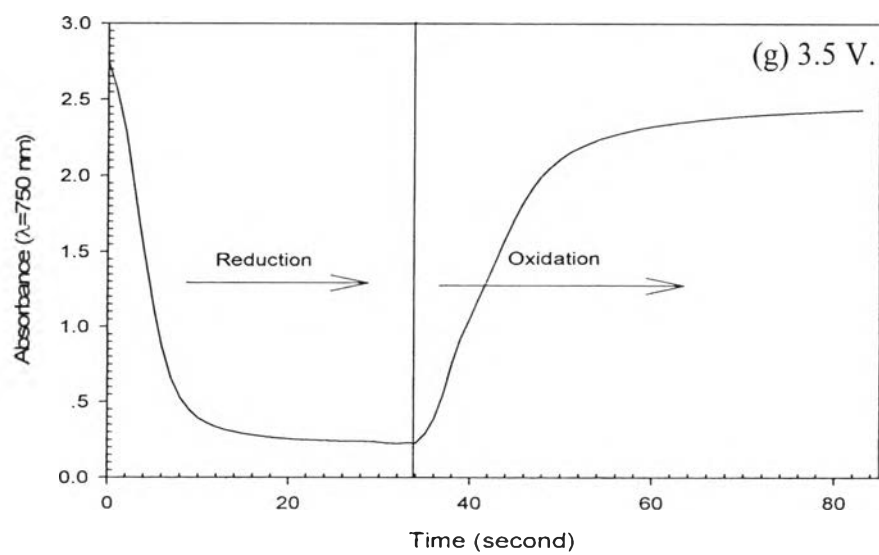
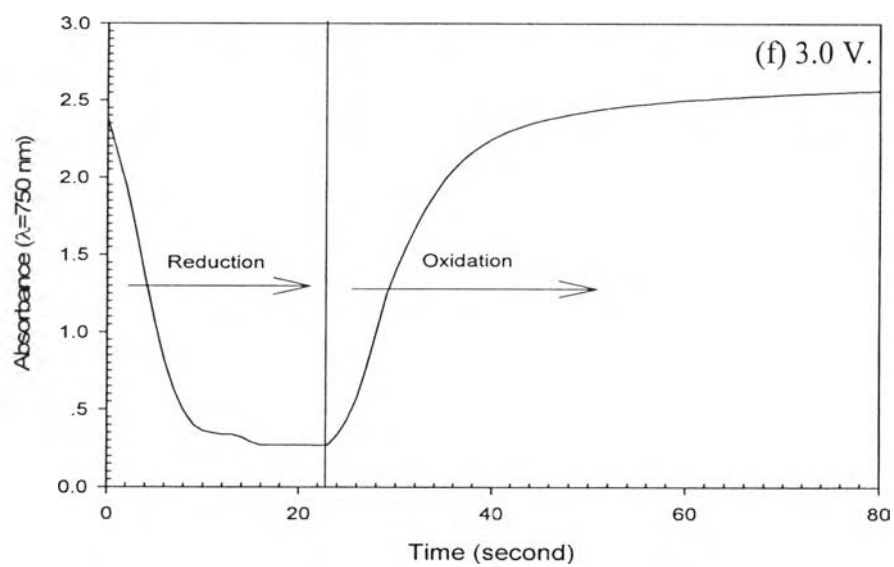
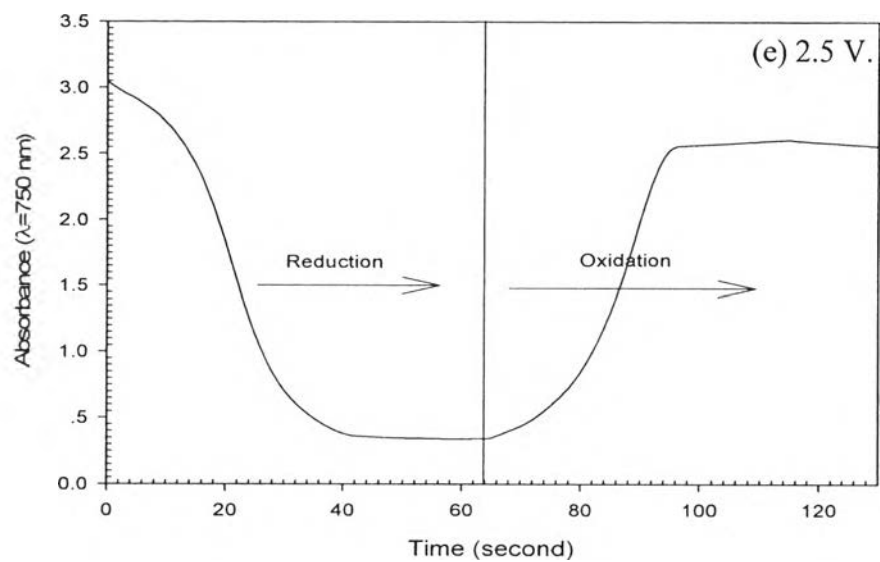
Table E8 The response times of PDMA/ITO synthesized in nitric acid for 3 minutes with the testing potential 1.5 V in the repeating experiments

Testing times at 1.5 V of PDMA films	Response time (sec)		
	reduction	oxidation	total
1 st	170	50	220
2 nd	179	60	239
3 rd	187	57	244
Average	178.7 ± 8.5	55.7 ± 5.13	234.3 ± 12.66

The response times of PDMA synthesized at 6 minutes in nitric acid at various potentials are shown in Figures E18 (a) to (j) and the data are tabulated in Table E9.







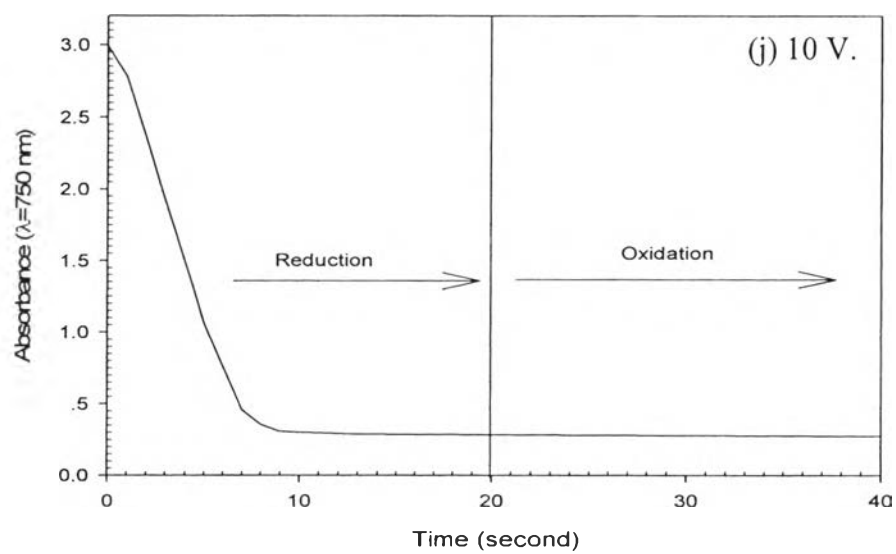
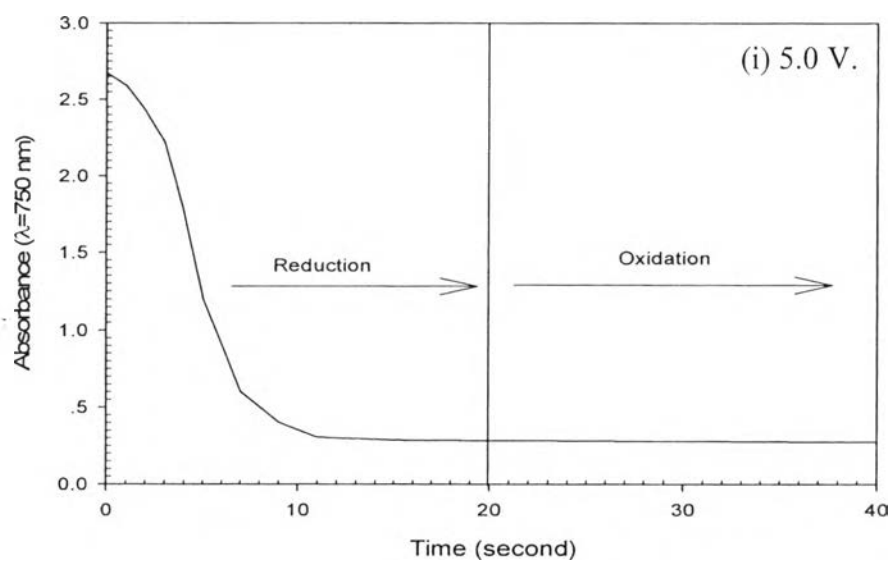
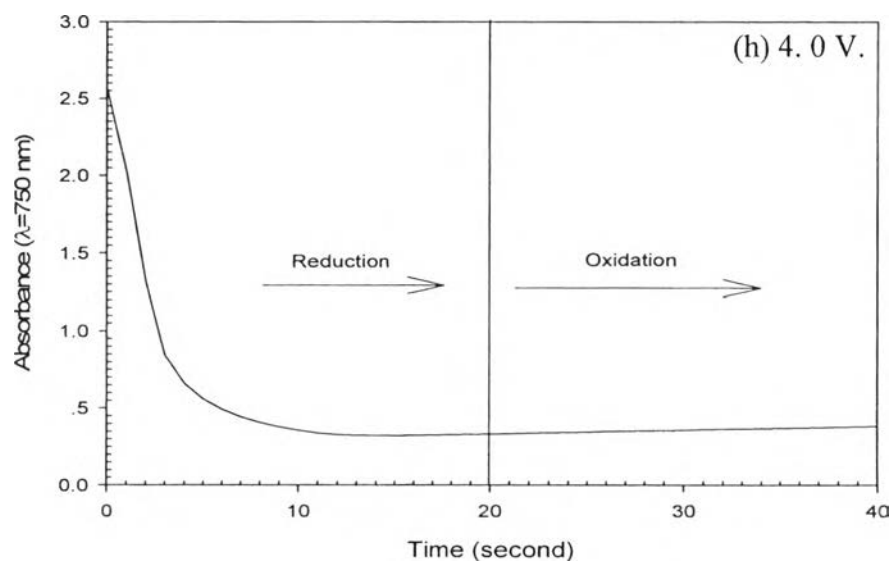


Figure E18 The response times of PDMA synthesized in nitric acid at 6 minutes with testing potentials (a) 0.5, (b) 1.0, (c) 1.5, (d) 2.0, (e) 2.5, (f) 3.0, (g) 3.5, (h) 4.0, (i) 5.0, and (j) 10 V via UV-Visible spectrophotometer.

Table E9 The reduction, oxidation and total response times of PDMA synthesized in nitric acid for 6 minutes at various testing potentials

Testing potential (V)	Reduction time (sec)	Oxidation time (sec)	Total response time (sec)
0.5	-	-	-
1.0	325	270	515
1.5	181	174	355
2.0	60	105	165
2.5	40	30	70
3.0	15	27	42
3.5	14	24	38
4.0	11	-	-
5.0	9.5	-	-
10	8.5	-	-

At the potential 1.5 V., the kinetic experiment was repeated 3 times in order to determine the average and the standard deviation (SD) values of the response time of PDMA/ITO synthesized for 6 minutes in nitric acid, as the results shown in Figure E19 and the response times are tabulated in Table E10.

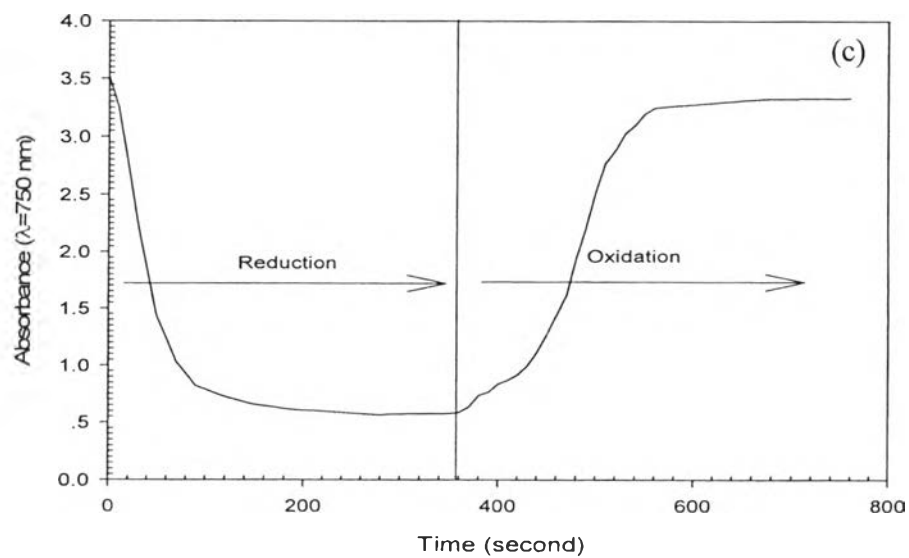
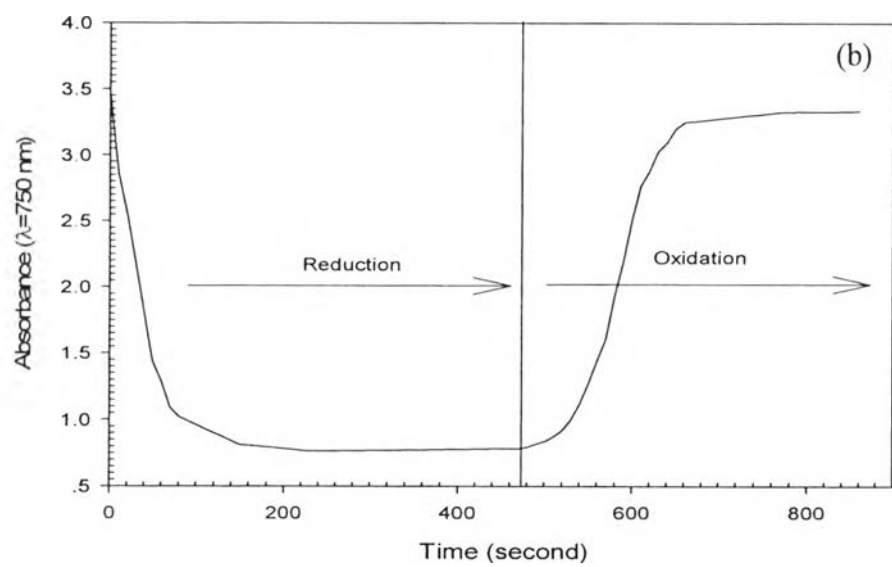
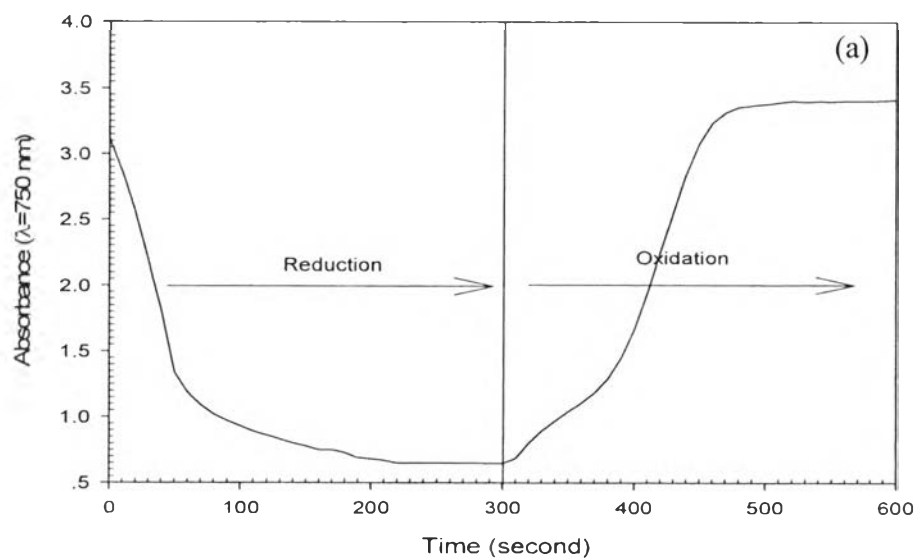
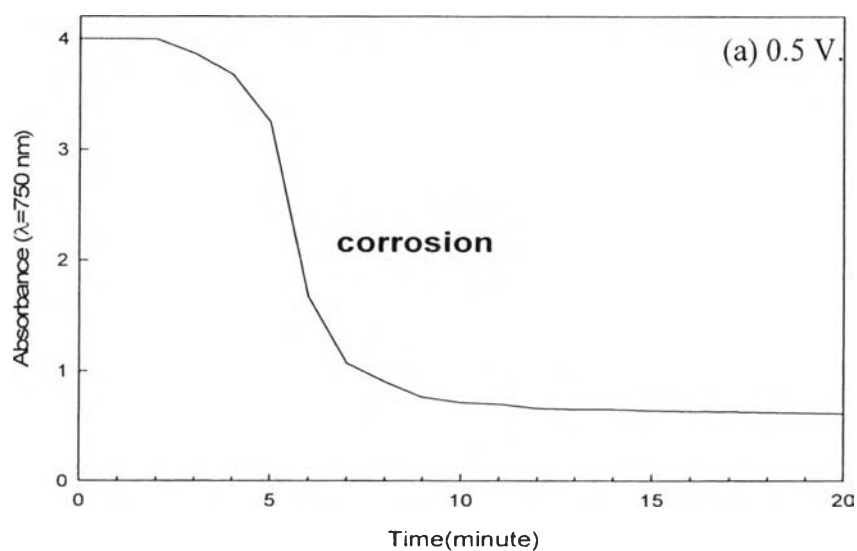


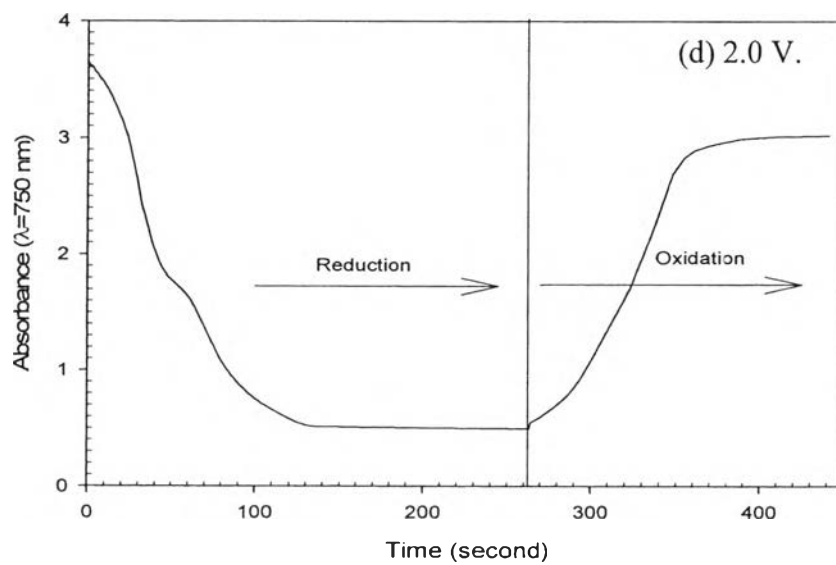
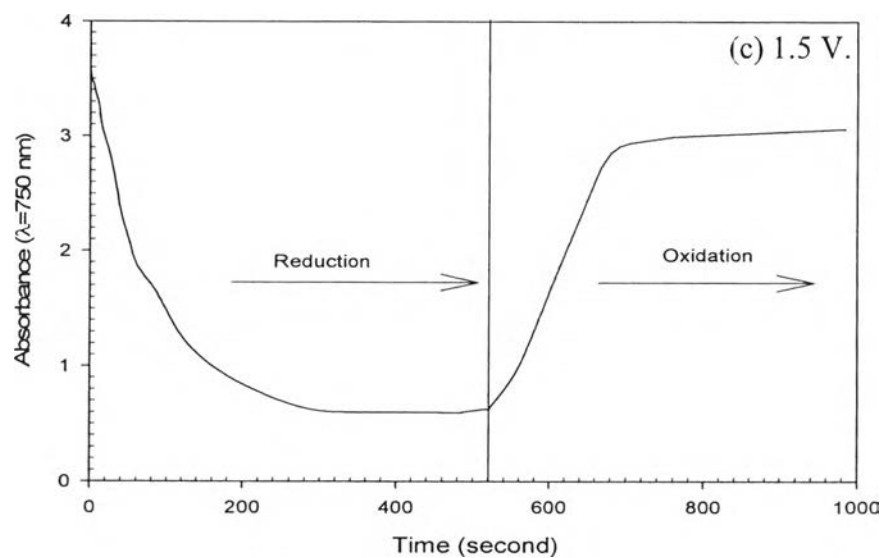
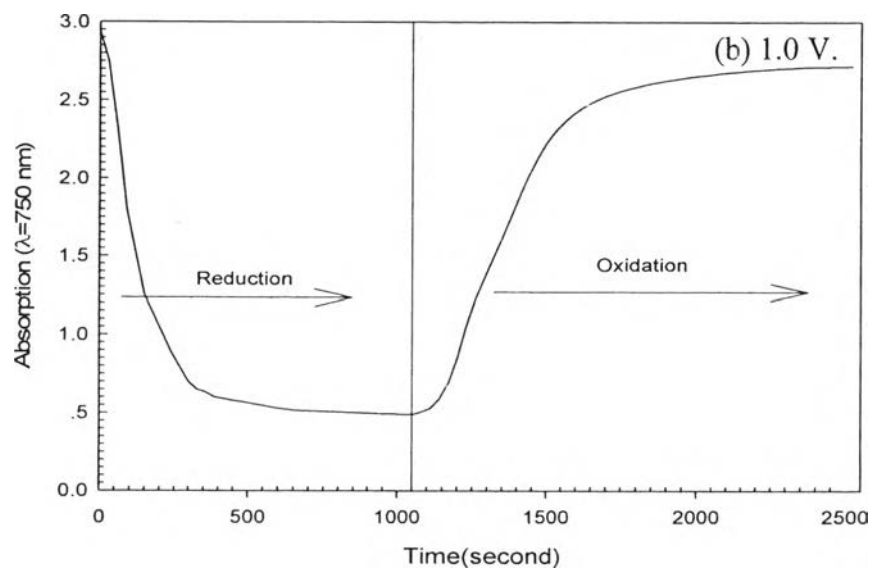
Figure E19 The response times of PDMA synthesized in nitric acid at 6 minutes with 3 times repeating experiments (a) to (c) at testing potential 1.5 V.

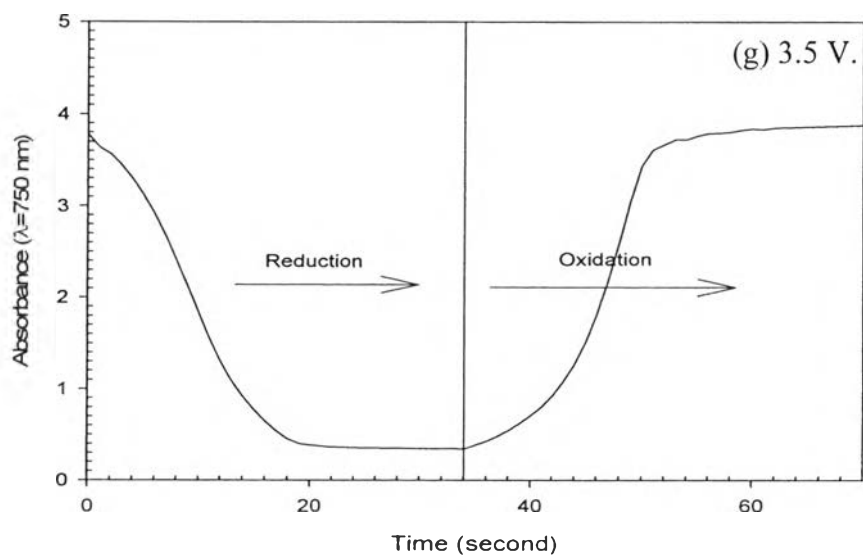
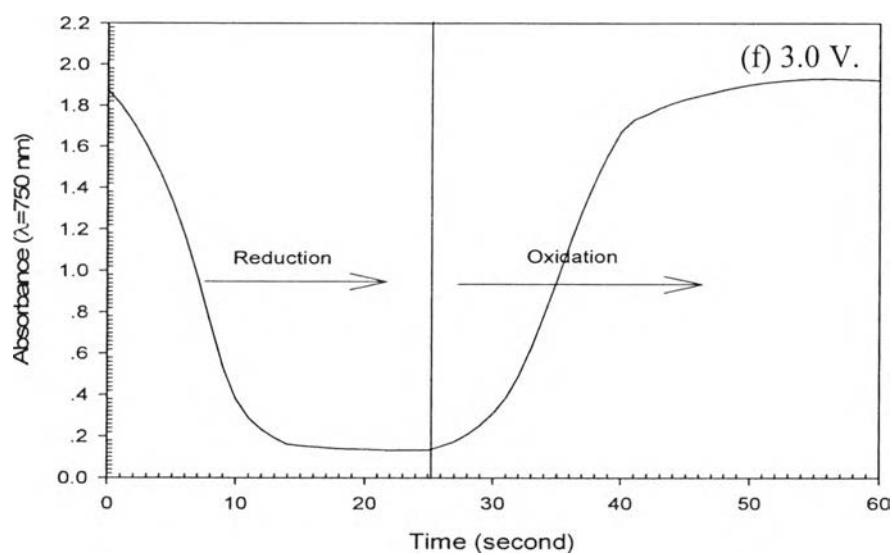
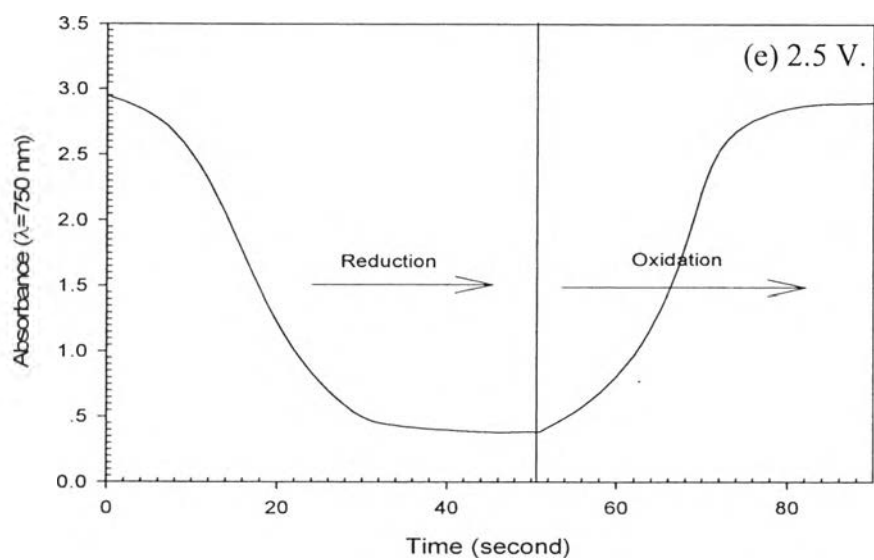
Table E10 The response times of PDMA/ITO synthesized in nitric acid for 6 minutes with testing potential 1.5 V in the repeating experiments

Testing times at 1.5 V of PDMA films	Response time (sec)		
	reduction	oxidation	total
1 st	190	160	340
2 nd	170	180	360
3 rd	178	190	368
Average	181.3 ± 6.2	174 ± 12.16	356 ± 14.4

The response time of PDMA synthesized at 10 minutes in nitric acid at various potentials are shown in Figures E20 (a) to (j) and the data are tabulated in Table E11.







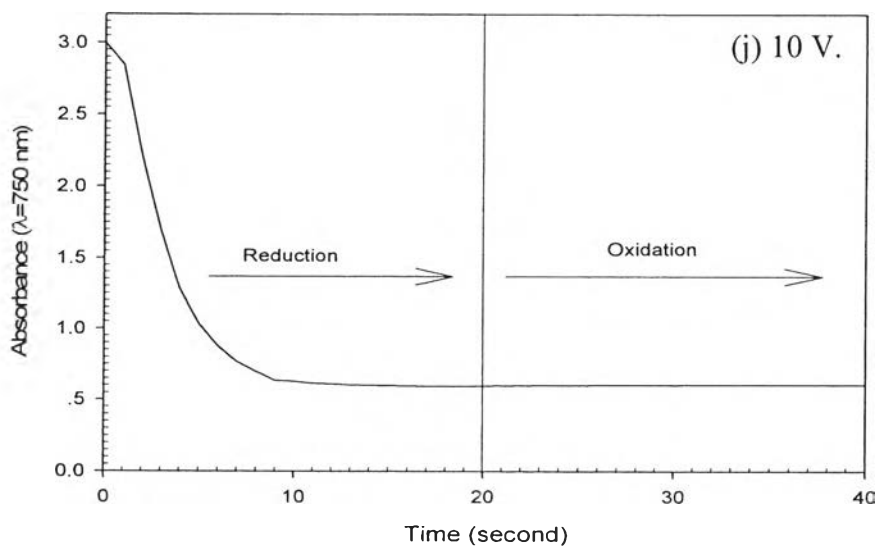
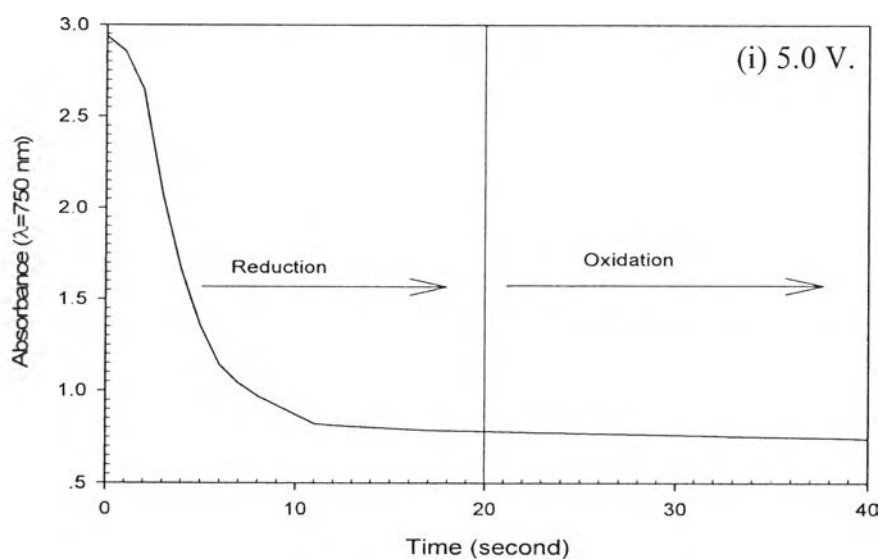
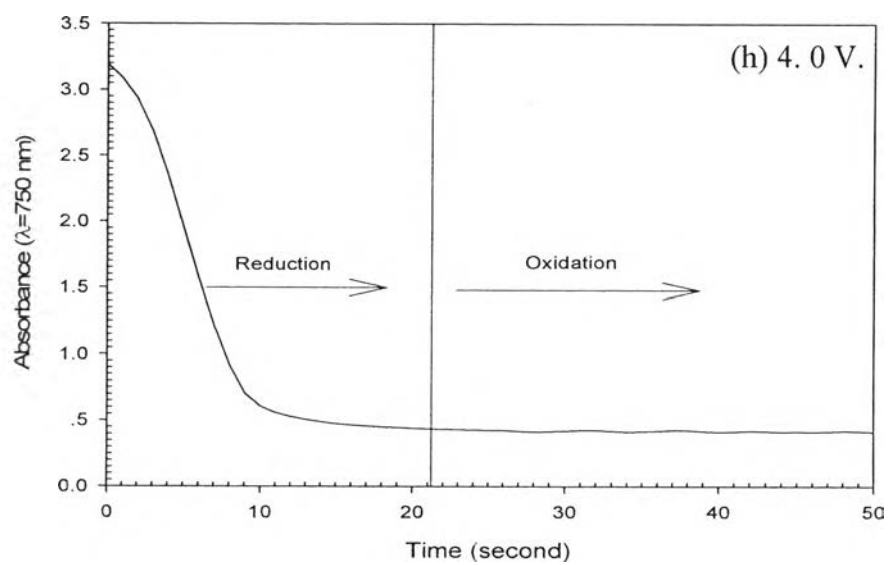


Figure E20 The response times of PDMA synthesized in nitric acid at 10 minutes with testing potentials (a) 0.5, (b) 1.0, (c) 1.5, (d) 2.0, (e) 2.5, (f) 3.0, (g) 3.5, (h) 4.0, (i) 5.0, and (j) 10 V via UV-Visible spectrophotometer.

Table E11 The reduction, oxidation and total response times of PDMA synthesized in nitric acid for 10 minutes at various testing potentials

Testing potential (V)	Reduction time (sec)	Oxidation time (sec)	Total response time (sec)
0.5	-	-	-
1.0	600	710	1310
1.5	267	167	434
2.0	130	105	245
2.5	34	22	56
3.0	18	20	38
3.5	18	17	35
4.0	13	-	-
5.0	10.8	-	-
10	8.9	-	-

At the potential 1.5 V., the kinetic experiment was repeated 3 times in order to determine the average and the standard deviation (SD) values of the response time of PDMA/ITO synthesized for 10 minutes in nitric acid, the results are shown in Figure E21 and the response times are tabulated in Table E12.

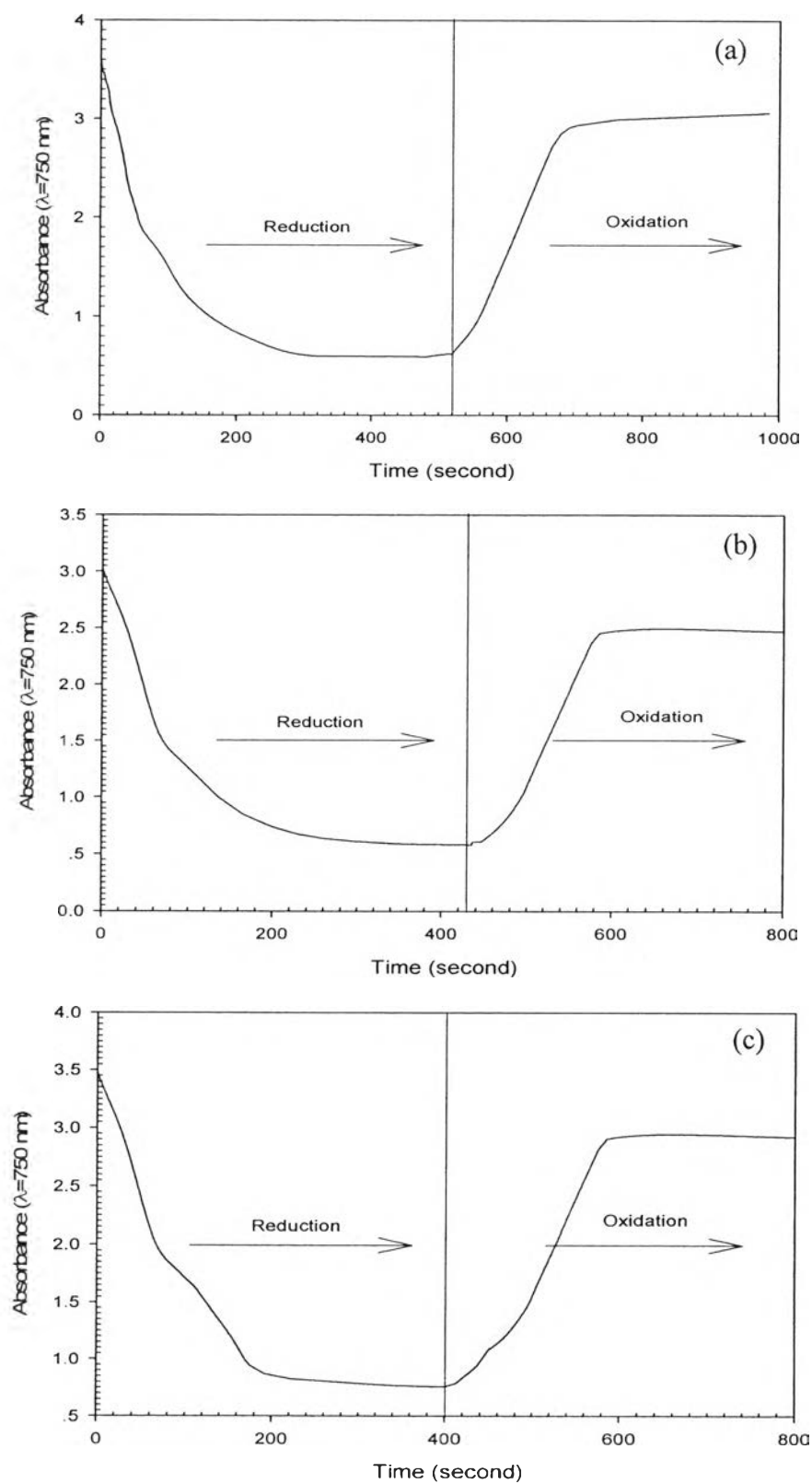


Figure E21 The response times of PDMA synthesized in nitric acid at 10 minutes with 3 times repeating experiments (a) to (c) at testing potential 1.5 V.

Table E12 The response times of PDMA/ITO synthesized in nitric acid for 10 minutes with testing potential 1.5 V in the repeating experiments

Testing times at 1.5 V of PDMA films	Response time (sec)		
	reduction	oxidation	total
1 st	270	170	440
2 nd	200	150	450
3 rd	250	180	430
Average	267 ± 15.3	167 ± 15.3	434 ± 5.8

The effect of the testing potentials on the reduction, oxidation and response times of PDMA synthesized in nitric acid at various dipping times are shown in Figures E22-E24.

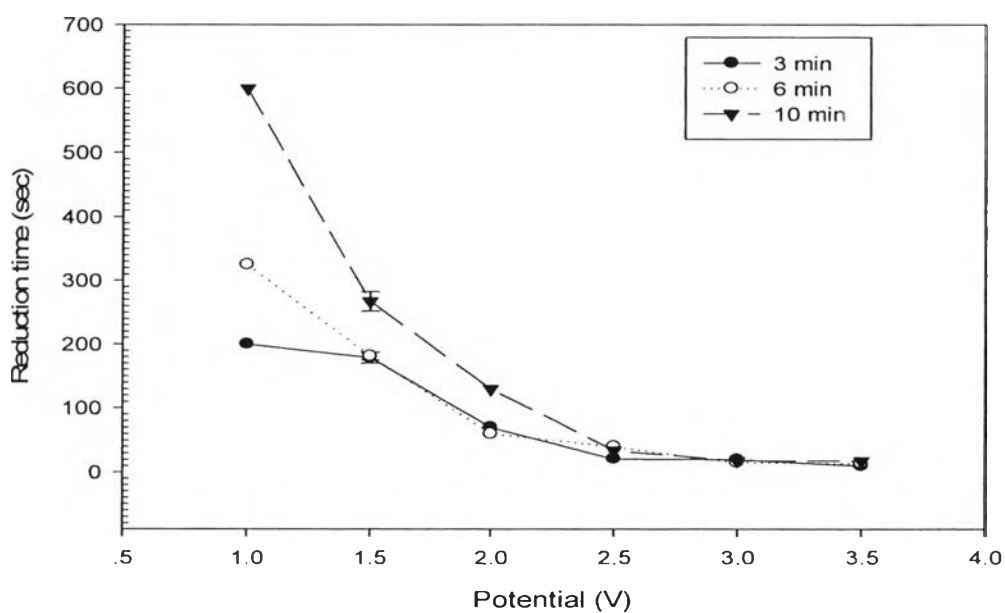


Figure E22 The effect of the testing potentials on the reduction time of PDMA synthesized in nitric acid at various dipping times.

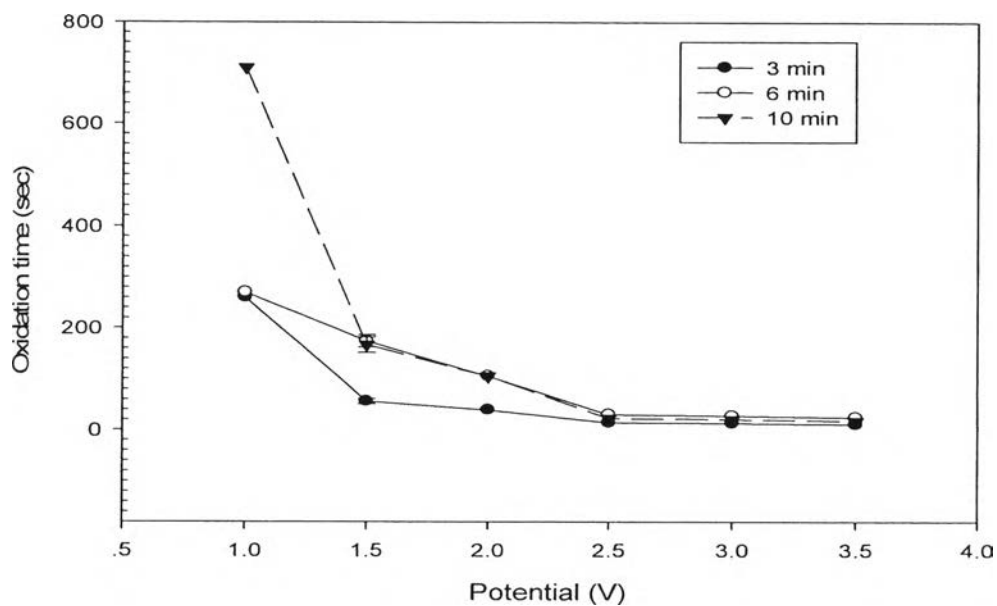


Figure E23 The effect of the testing potentials on the oxidation time of PDMA synthesized in nitric acid at various dipping times.

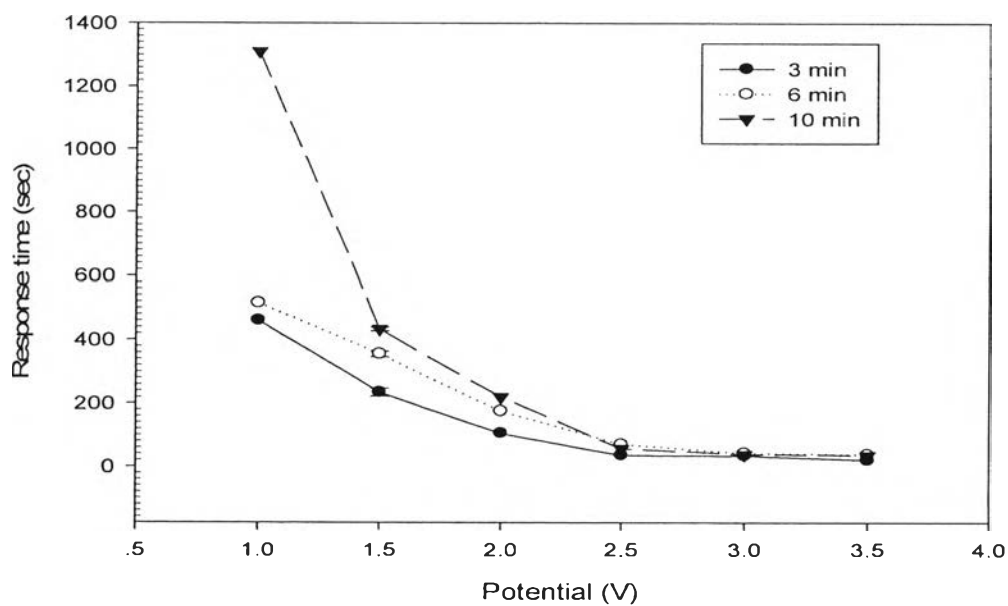
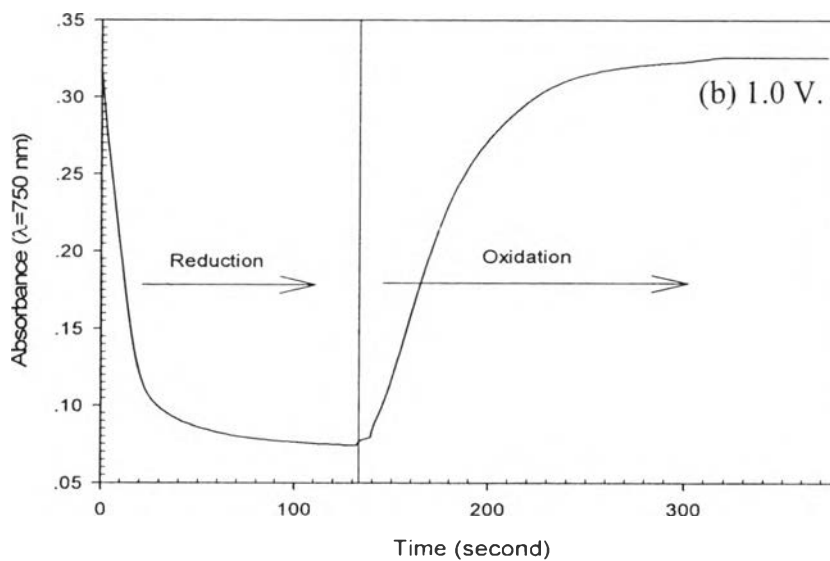
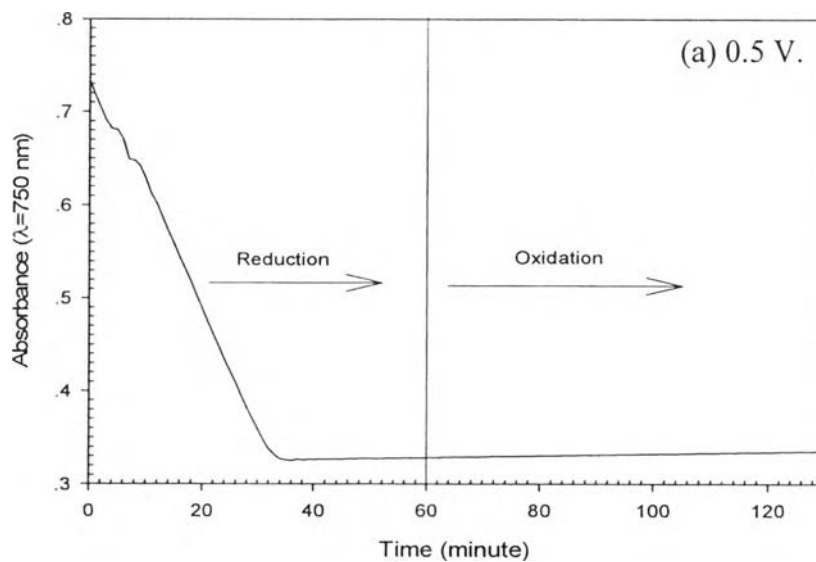
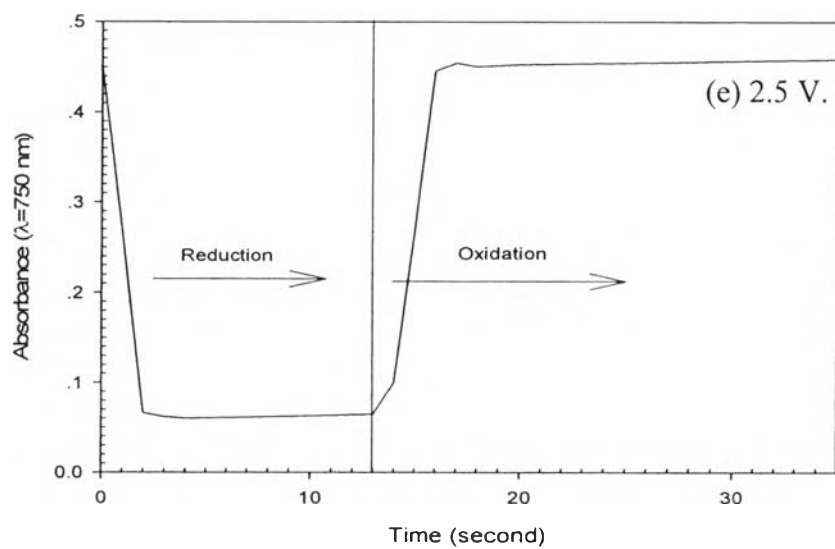
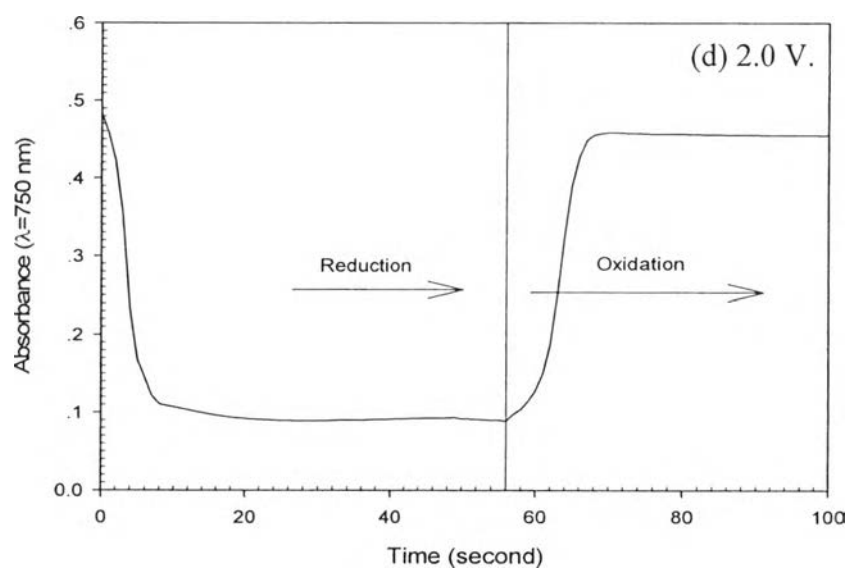
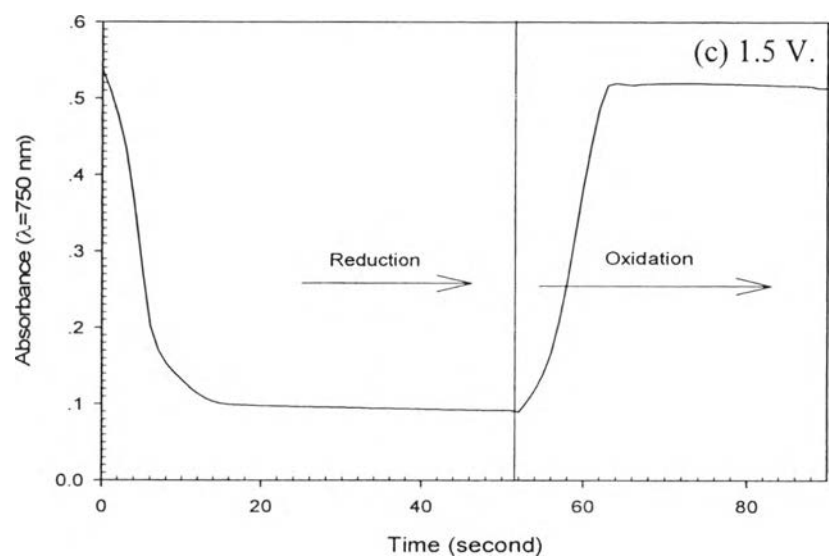
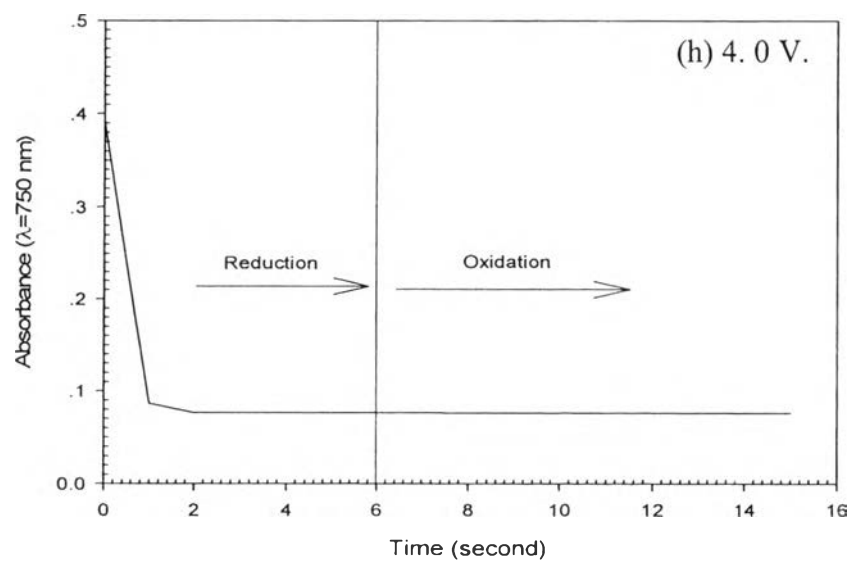
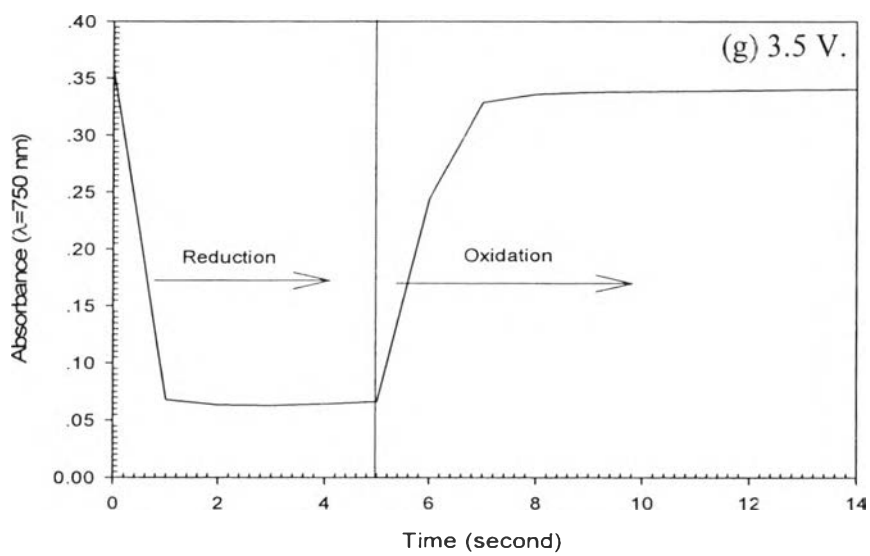
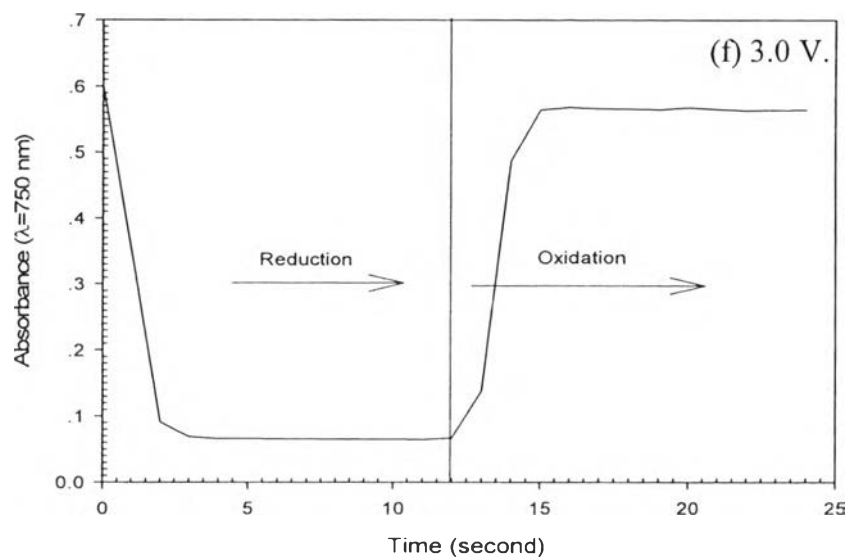


Figure E24 The effect of the testing potentials on the response time of PDMA synthesized in nitric acid at various dipping times.

The response times of PDMA synthesized at 6 minutes in hydrochloric acid at various potentials are shown in Figures E25 (a) to (j) and the data are tabulated in Table E13.







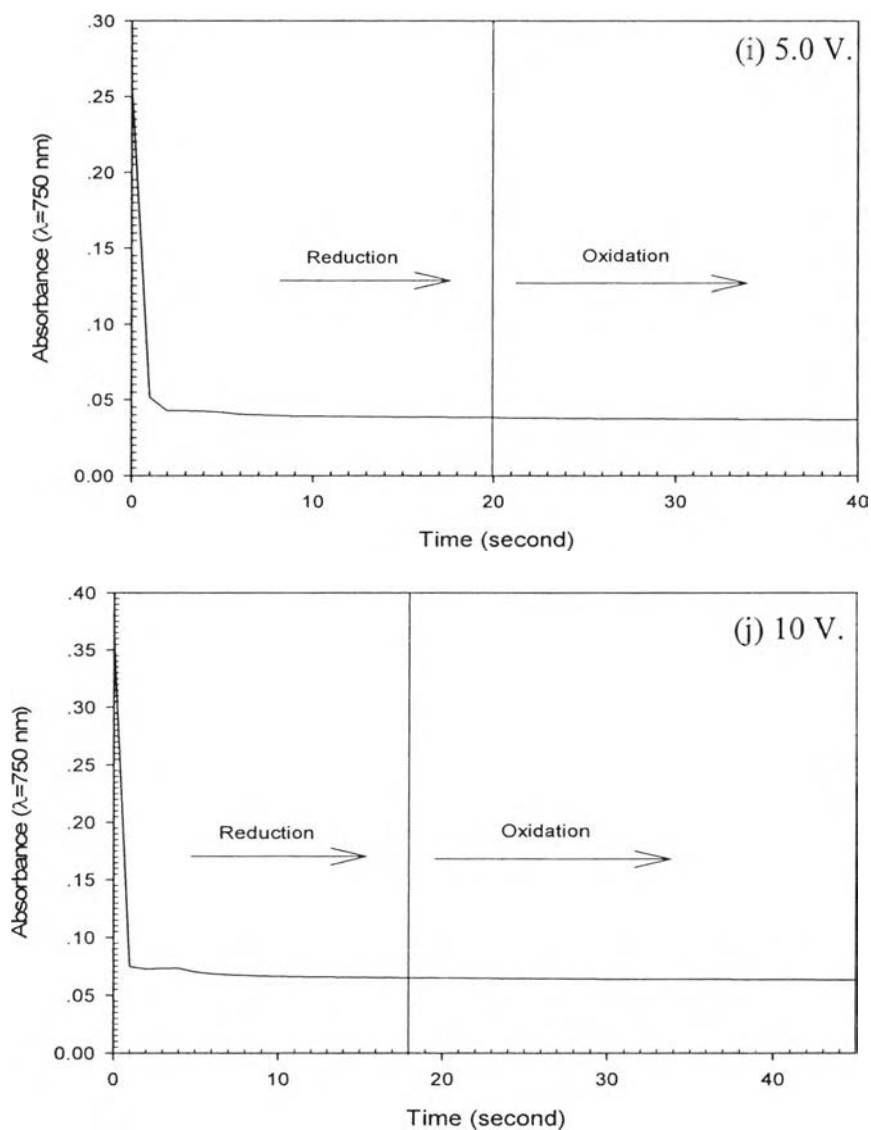
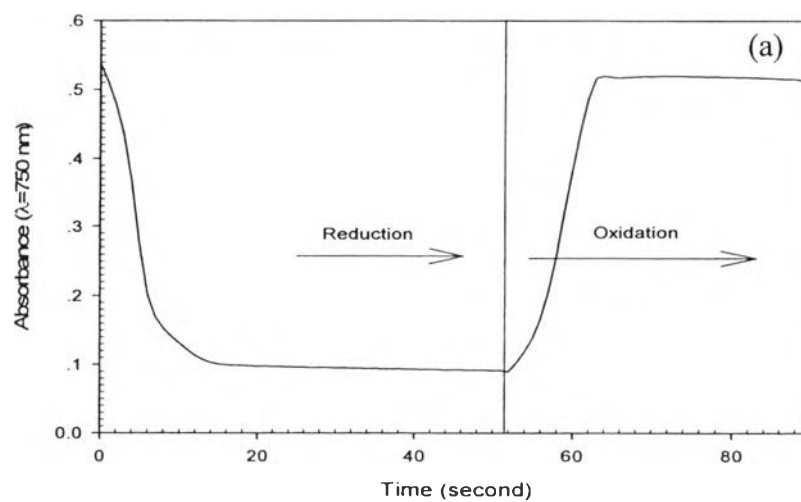


Figure E25 The response times of PDMA synthesized in hydrochloric acid at 6 minutes with testing potentials (a) 0.5, (b) 1.0, (c) 1.5, (d) 2.0, (e) 2.5, (f) 3.0, (g) 3.5, (h) 4.0, (i) 5.0, and (j) 10 V via UV-Visible spectrophotometer.

Table E13 The reduction, oxidation and total response times of PDMA synthesized in hydrochloric acid for 6 minutes at various testing potentials

Testing potential (V)	Reduction time (sec)	Oxidation time (sec)	Total response time (sec)
0.5	1920	-	-
1.0	78	104	182
1.5	17.7	10.7	28.4
2.0	17	11	28
2.5	10	2.7	12.7
3.0	3	2.8	5.8
3.5	1.7	2	3.7
4.0	1.5	-	-
5.0	1.4	-	-
10	1.0	-	-

At the potential 1.5 V., the kinetic experiment was repeated 3 times in order to determine the average and the standard deviation (SD) values of the response time of PDMA/ITO synthesized for 6 minutes in hydrochloric acid, the results are shown in Figure E26 and the response times are tabulated in Table E14.



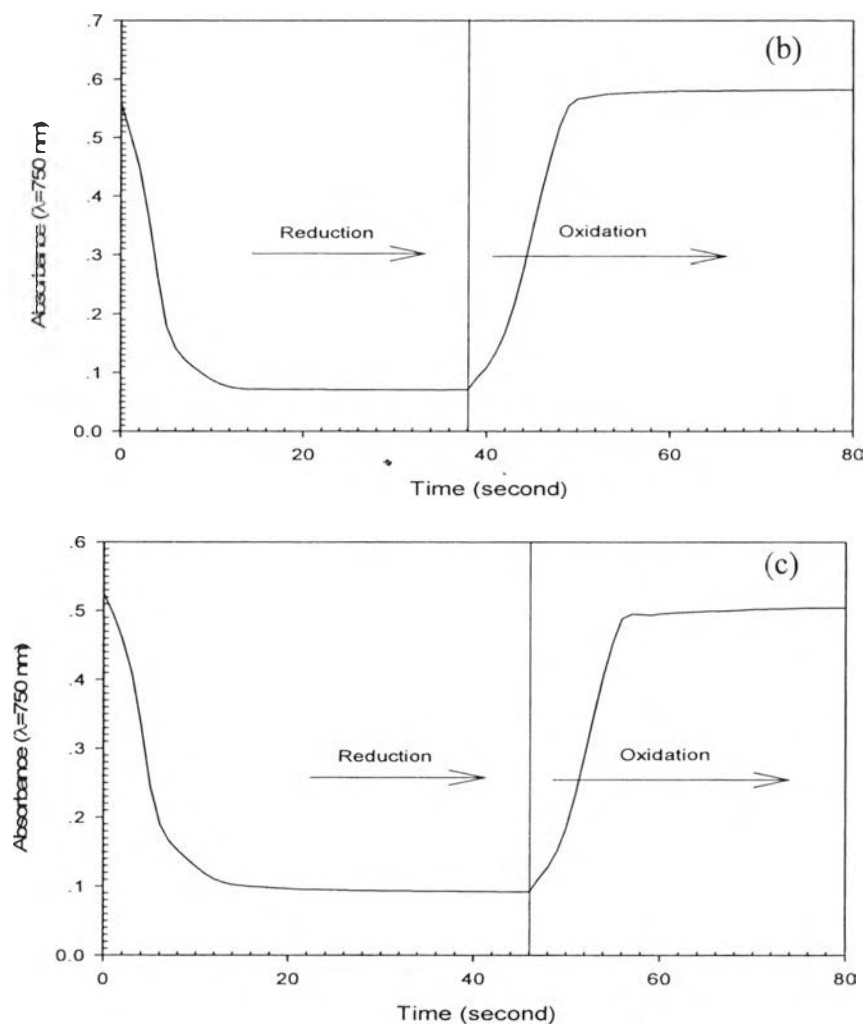
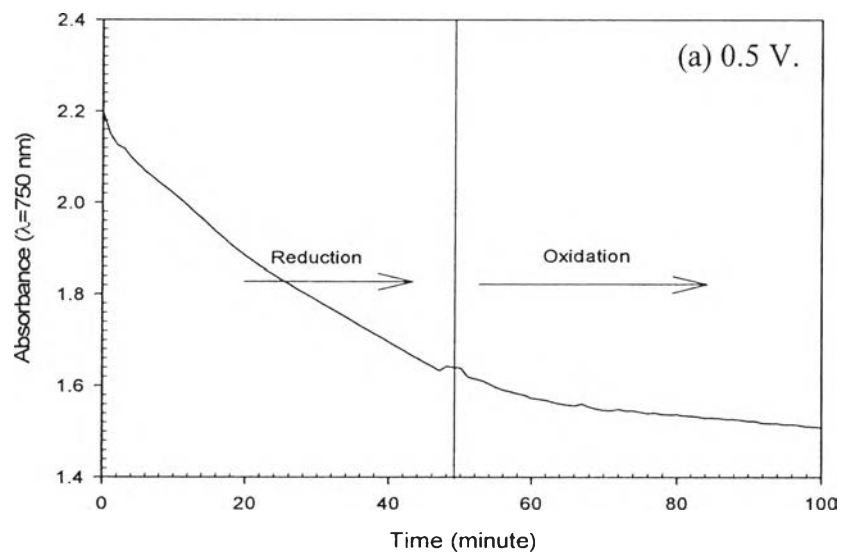


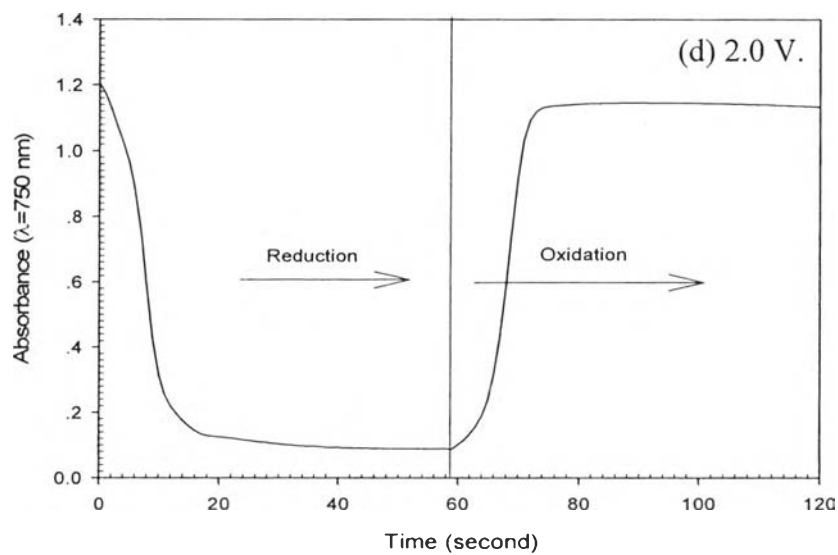
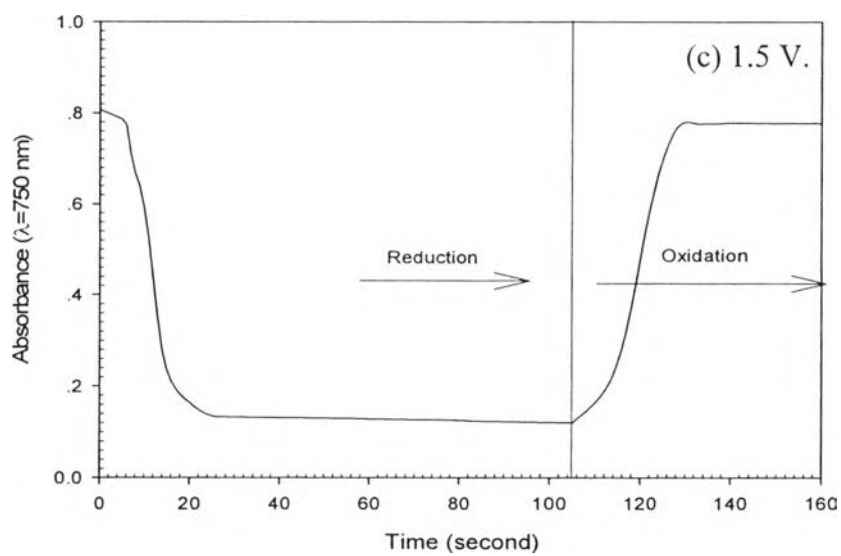
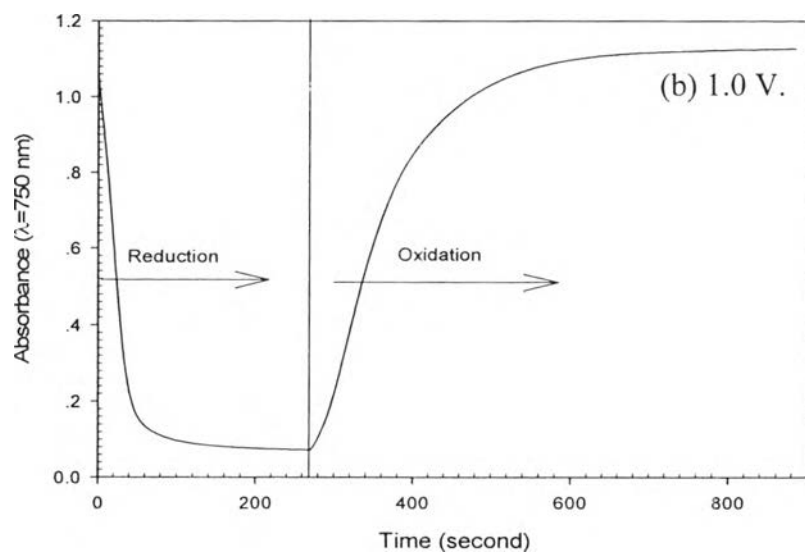
Figure E26 The response times of PDMA synthesized in hydrochloric acid at 6 minutes with 3 times repeating experiments (a) to (c) at testing potential 1.5 V.

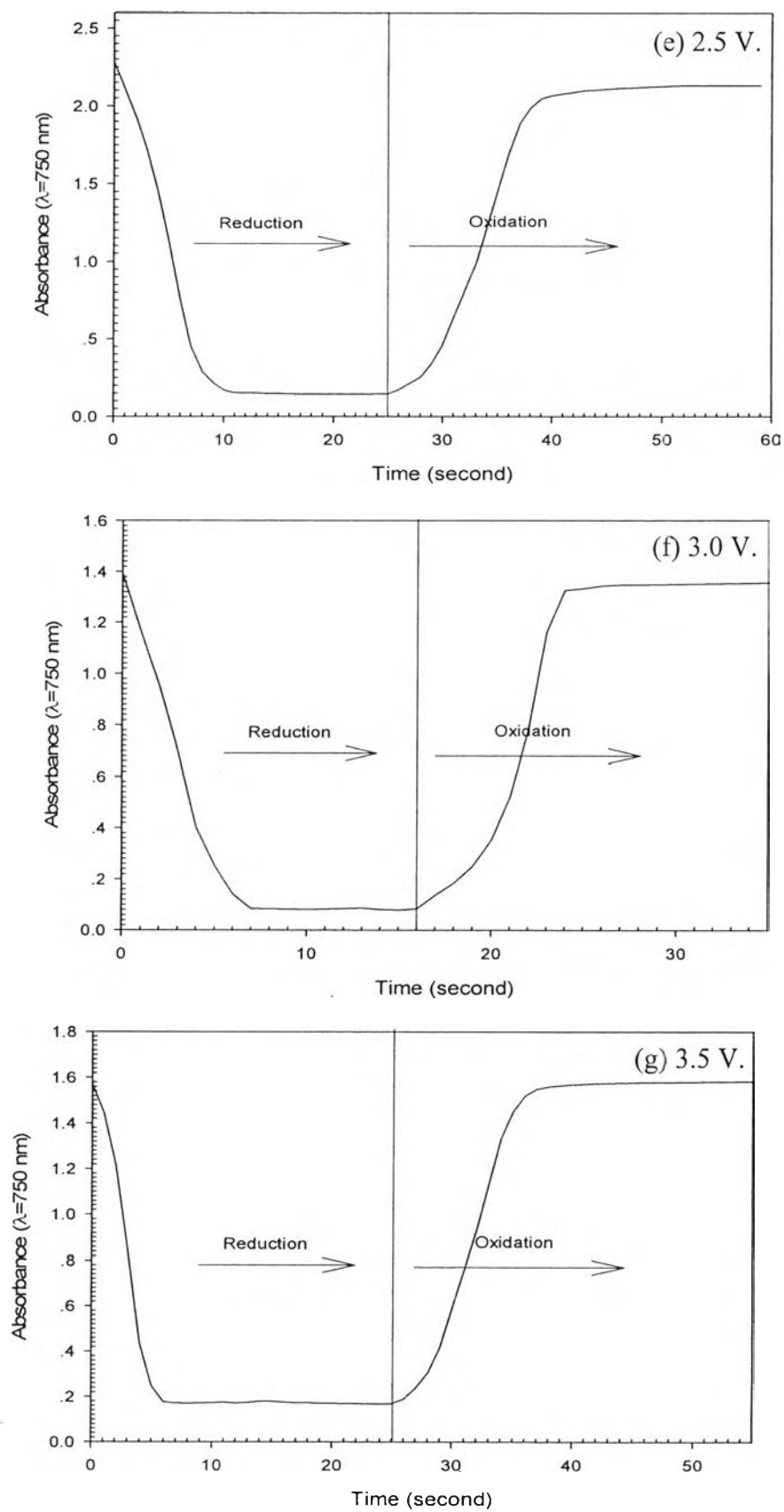
Table E14 The response times of PDMA/ITO synthesized in hydrochloric acid for 6 minutes with testing potential 1.5 V in the repeating experiments

Testing times at 1.5 V of PDMA films	Response time (sec)		
	reduction	oxidation	total
1 st	20	11	31
2 nd	18	11	29
3 rd	15	10	25
Average	17.7 ± 2.5	10.7 ± 0.6	28.3 ± 3.1

The response times of PDMA synthesized at 10 minutes in hydrochloric acid at various potentials are shown in Figures E27 (a) to (j) and the data are tabulated in Table E15.







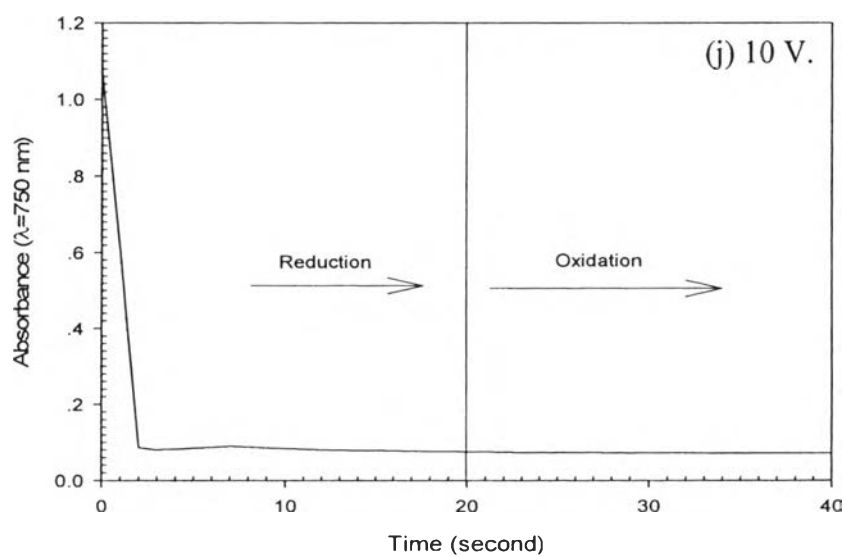
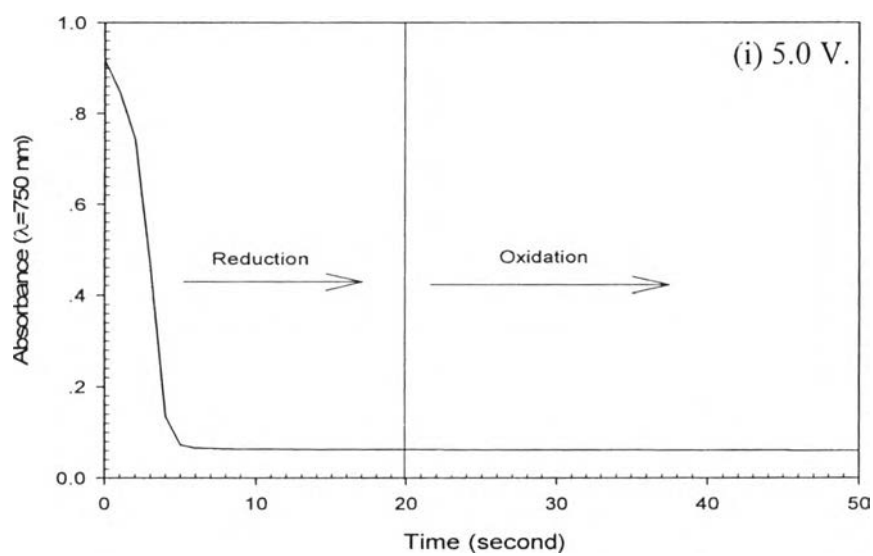
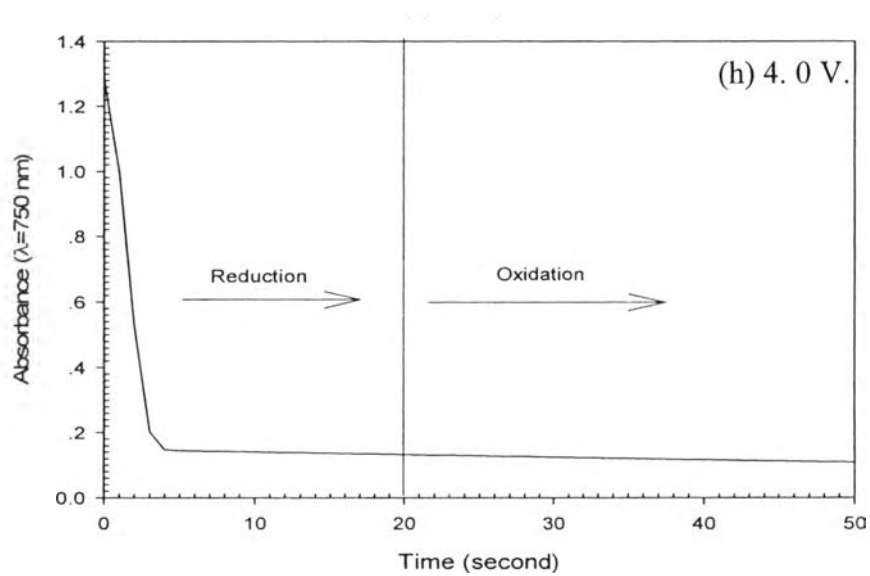


Figure E27 The response times of PDMA synthesized in hydrochloric acid at 10 minutes with testing potentials (a) 0.5, (b) 1.0, (c) 1.5, (d) 2.0, (e) 2.5, (f) 3.0, (g) 3.5, (h) 4.0, (i) 5.0, and (j) 10 V via UV-Visible spectrophotometer.

Table E15 The reduction, oxidation and total response times of PDMA synthesized in hydrochloric acid for 10 minutes at various testing potentials

Testing potential (V)	Reduction time (sec)	Oxidation time (sec)	Total response time (sec)
0.5	-	-	-
1.0	125	225	350
1.5	26.7	22	48.7
2.0	25	13	38
2.5	10	13	23
3.0	6.8	9	15.8
3.5	6	9	15
4.0	5	-	-
5.0	4.9	-	-
10	2.8	-	-

At the potential 1.5 V., the kinetic experiment was repeated 3 times in order to determine the average and the standard deviation (SD) values of the response time of PDMA/ITO synthesized for 10 minutes in hydrochloric acid, the results are shown in Figure E28 and the response times are tabulated in Table E16.

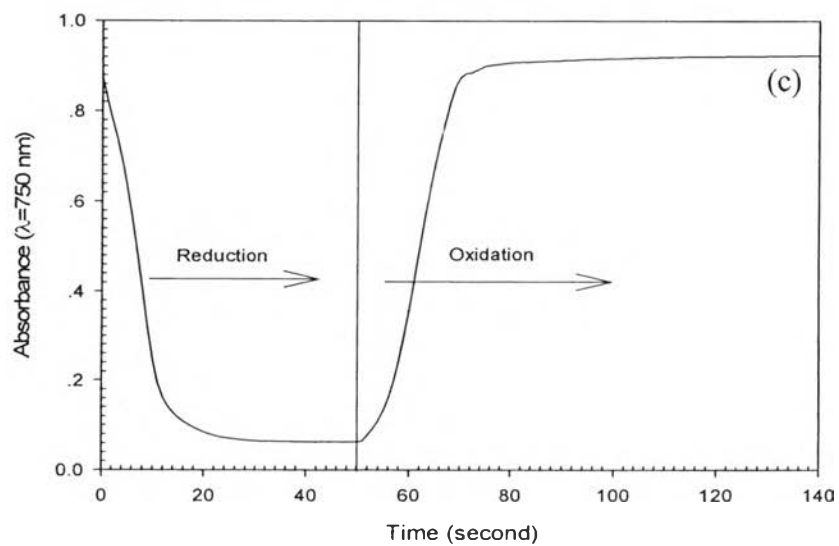
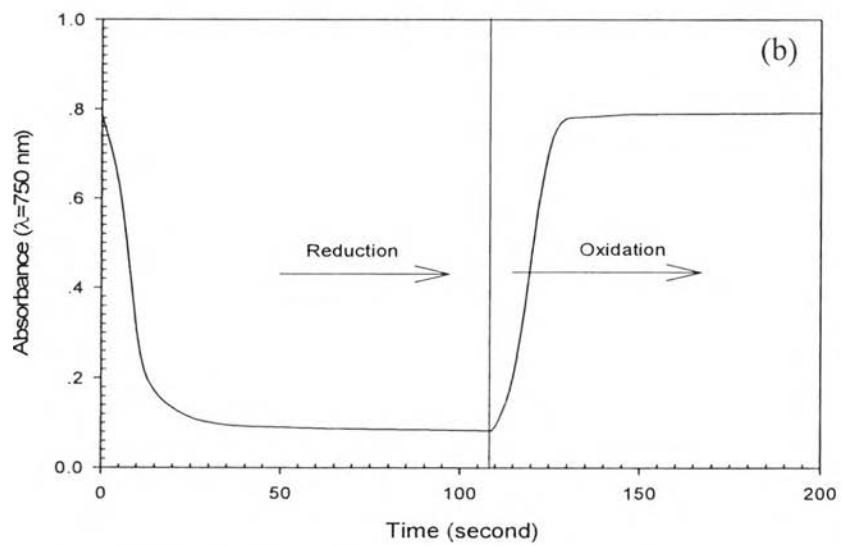
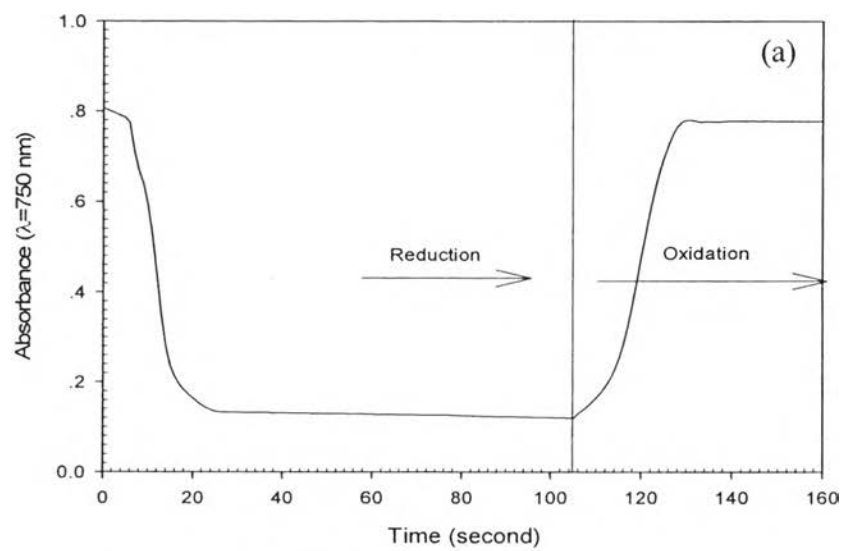


Figure E28 The response times of PDMA synthesized in hydrochloric acid at 10 minutes with 3 times repeating experiments (a) to (c) at testing potential 1.5 V.

Table E16 The response times of PDMA/ITO synthesized in hydrochloric acid for 10 minutes with testing potential 1.5 V in the repeating experiments

Testing times at 1.5 V of PDMA films	Response time (sec)		
	reduction	oxidation	total
1 st	27	21	48
2 nd	29	24	53
3 rd	24	21	45
Average	26.7 ± 2.5	22 ± 1.73	48.7 ± 2.52

The effect of the testing potentials on the reduction, oxidation and response times of PDMA synthesized in hydrochloric acid at various dipping times are shown in Figures E29-E31.

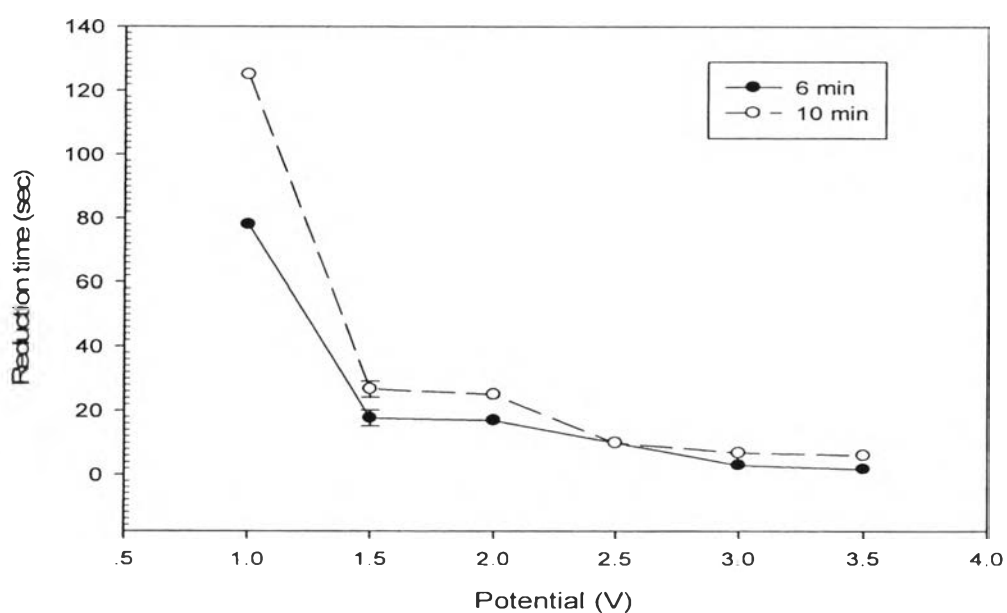


Figure E29 The effect of the testing potentials on the reduction time of PDMA synthesized in hydrochloric acid at various dipping times.

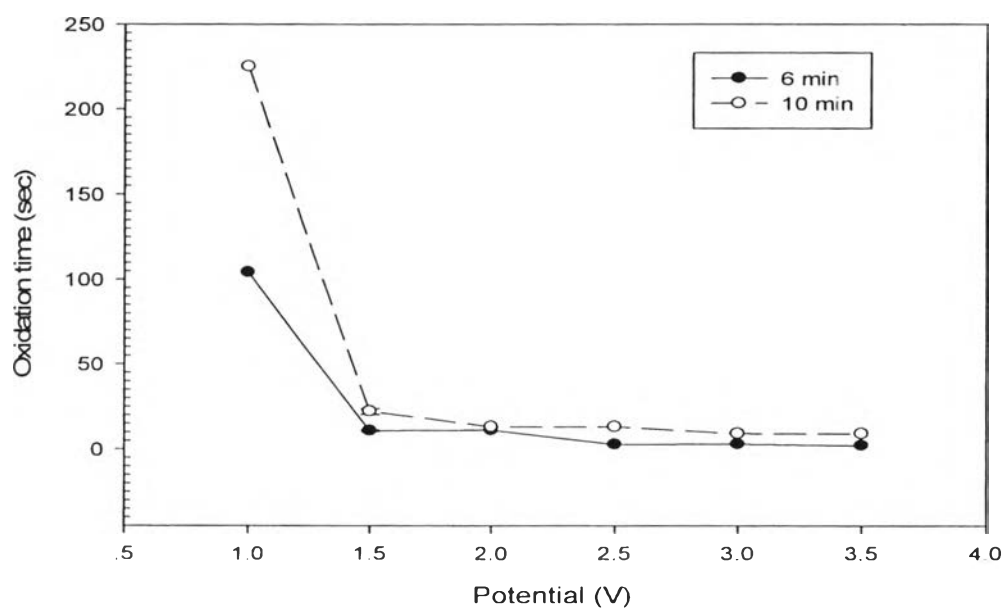


Figure E30 The effect of the testing potentials on the oxidation time of PDMA synthesized in hydrochloric acid at various dipping times.

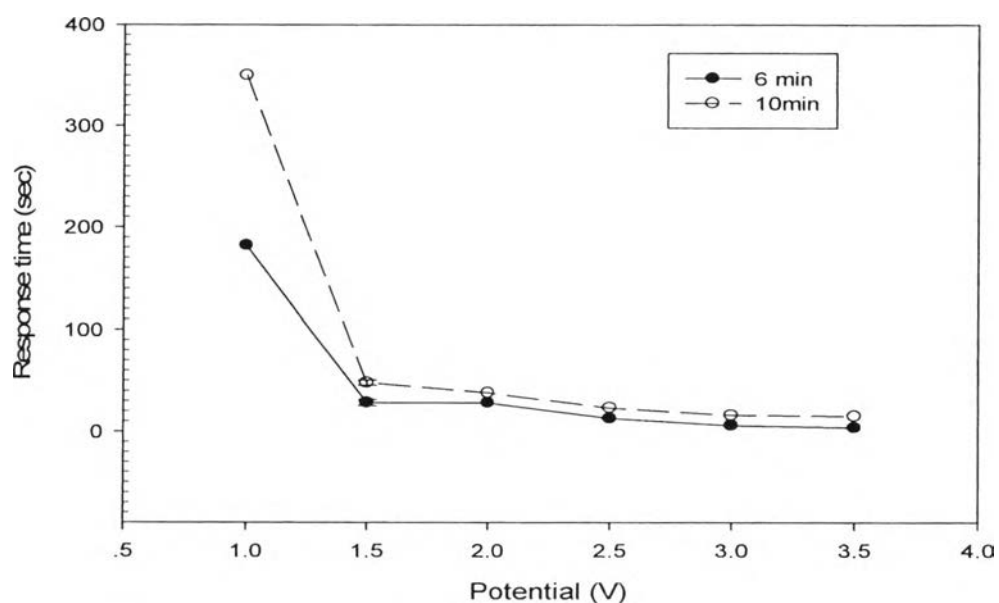
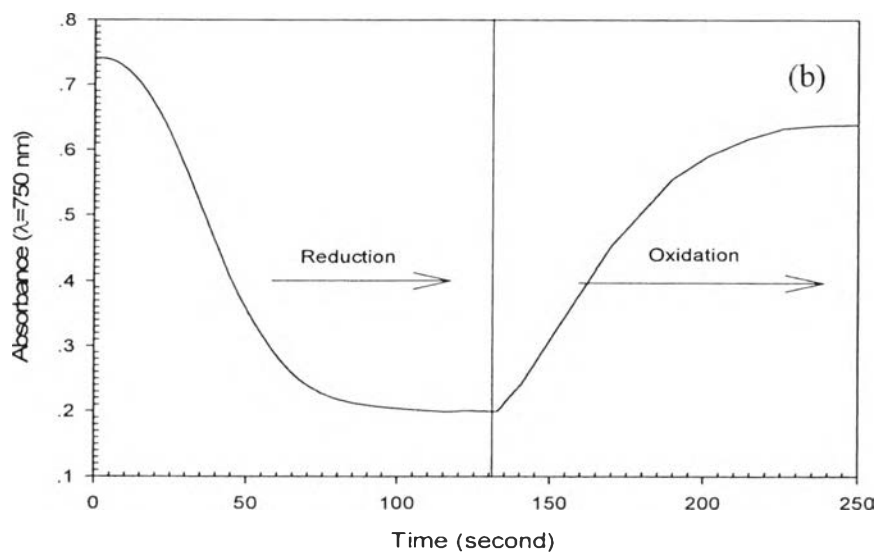
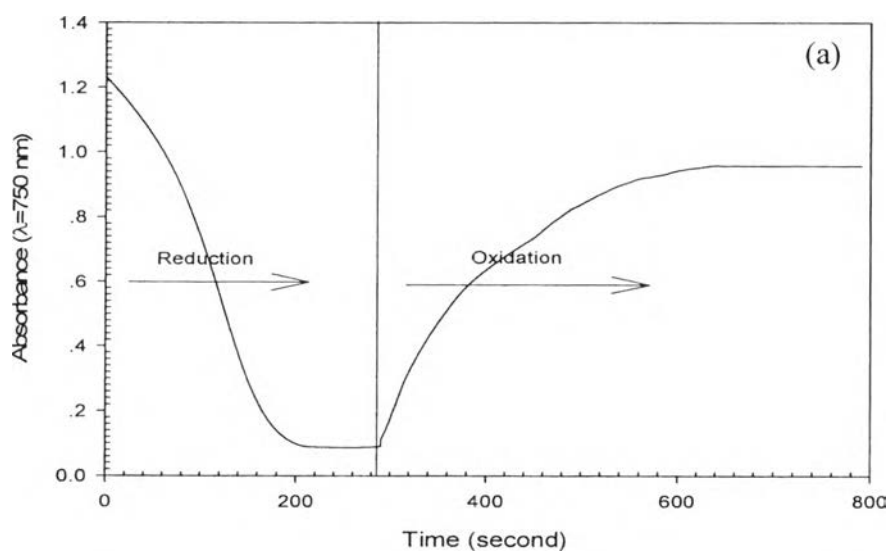
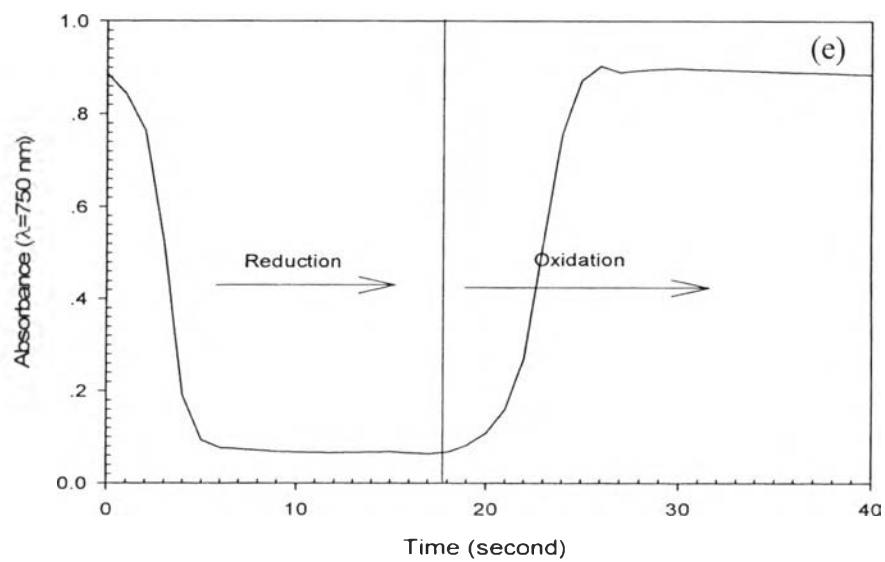
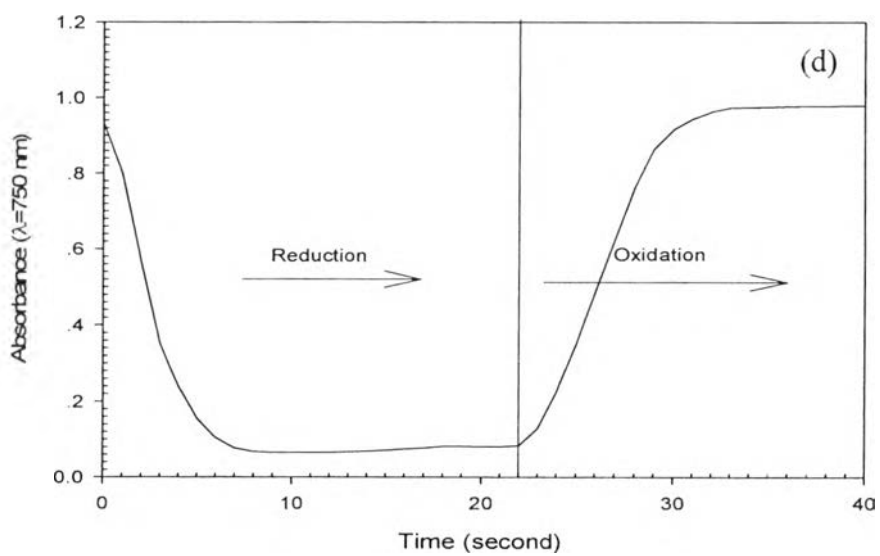
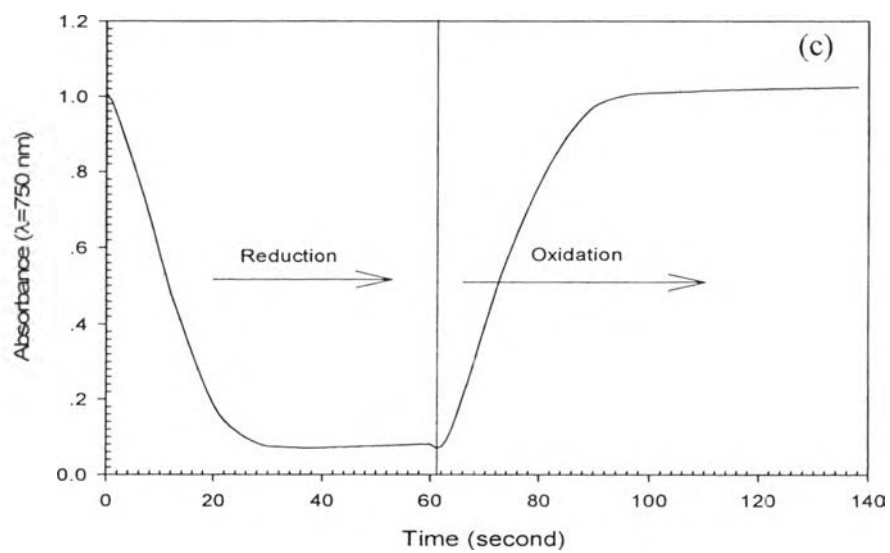


Figure E31 The effect of the testing potentials on the response time of PDMA synthesized in hydrochloric acid at various dipping times.

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 acid for 3 minutes were tested at 3.5 V in various concentrations of sulfuric acid (10^{-6} , 10^{-5} , 10^{-4} , 10^{-3} , 10^{-2} , and 10^{-1} M). The absorption curves are shown in Figures E32 (a) to (f), respectively.





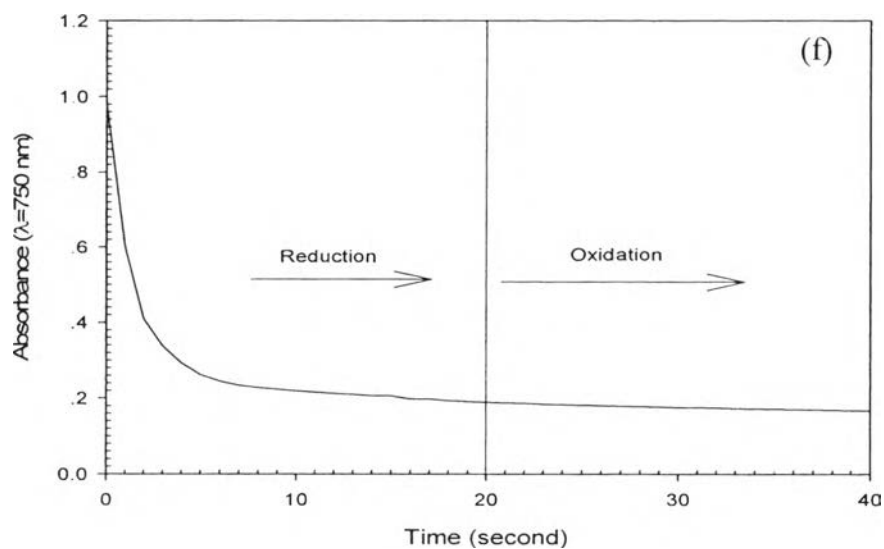


Figure E32 The absorption curves of PDMA synthesized in oxalic acid for 3 minutes tested at 3.5 V in (a) 10^{-6} , (b) 10^{-5} , (c) 10^{-4} , (d) 10^{-3} , (e) 10^{-2} , and (f) 10^{-1} M sulfuric acid.

The effect of sulfuric concentration on the reduction, oxidation, and response times of PDMA synthesized in oxalic acid at 3 minutes is shown in Figures E33 to E35, respectively.

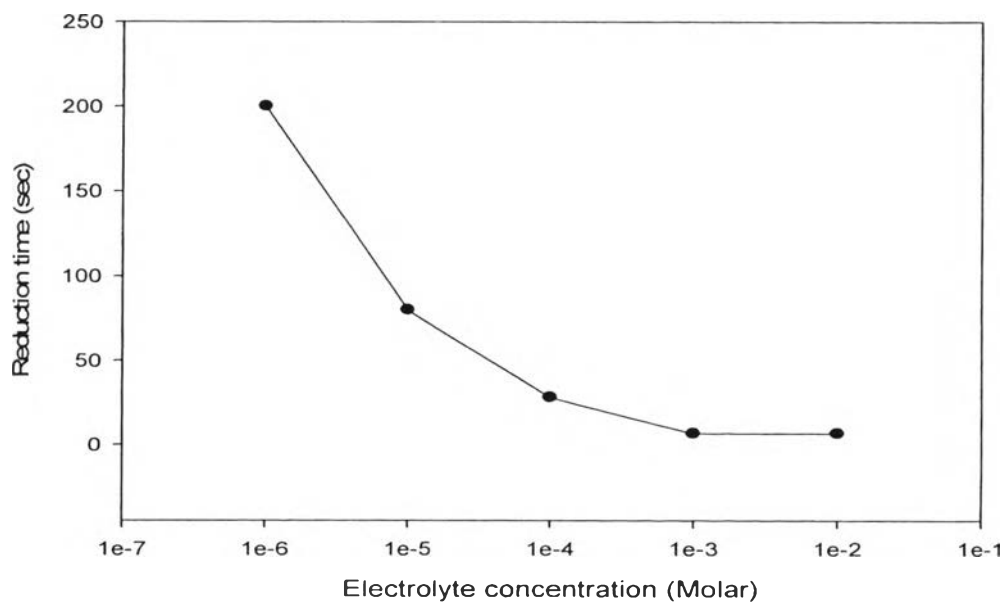


Figure E33 The effect of sulfuric concentration on the reduction time of PDMA synthesized in oxalic acid at 3 minutes.

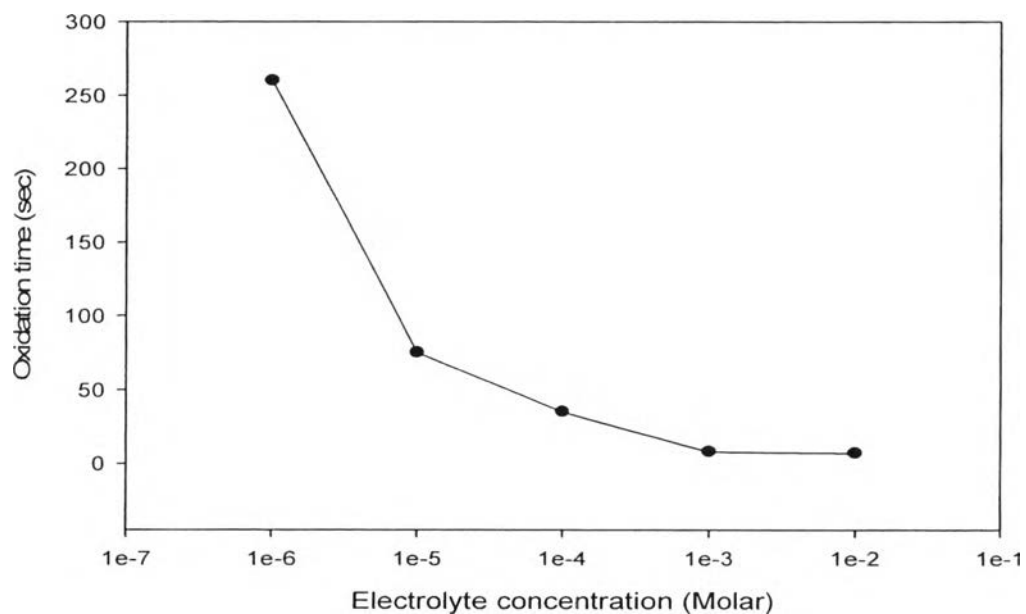


Figure E34 The effect of sulfuric concentration on the oxidation time of PDMA synthesized in oxalic acid at 3 minutes.

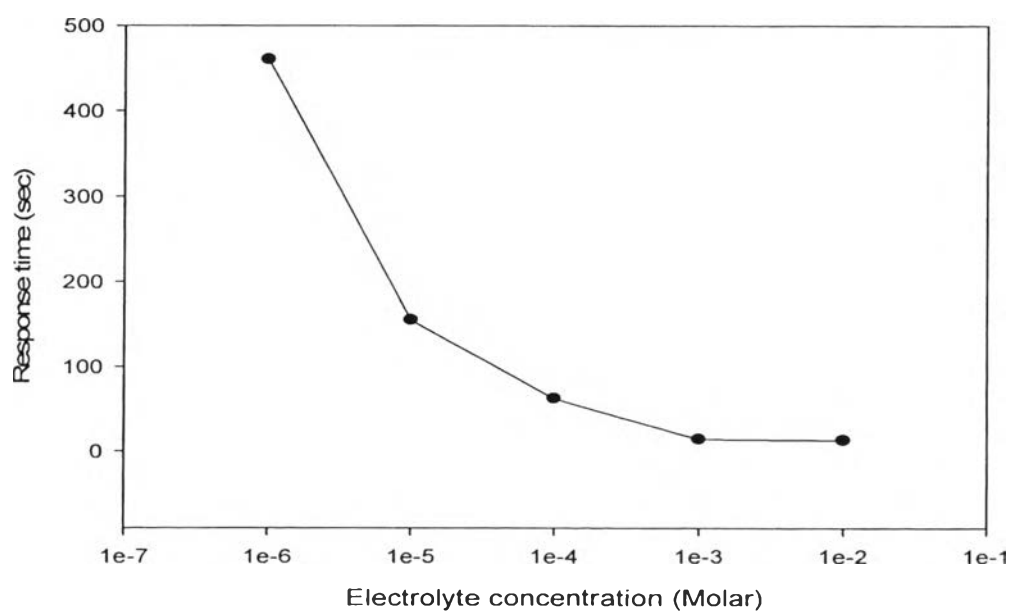
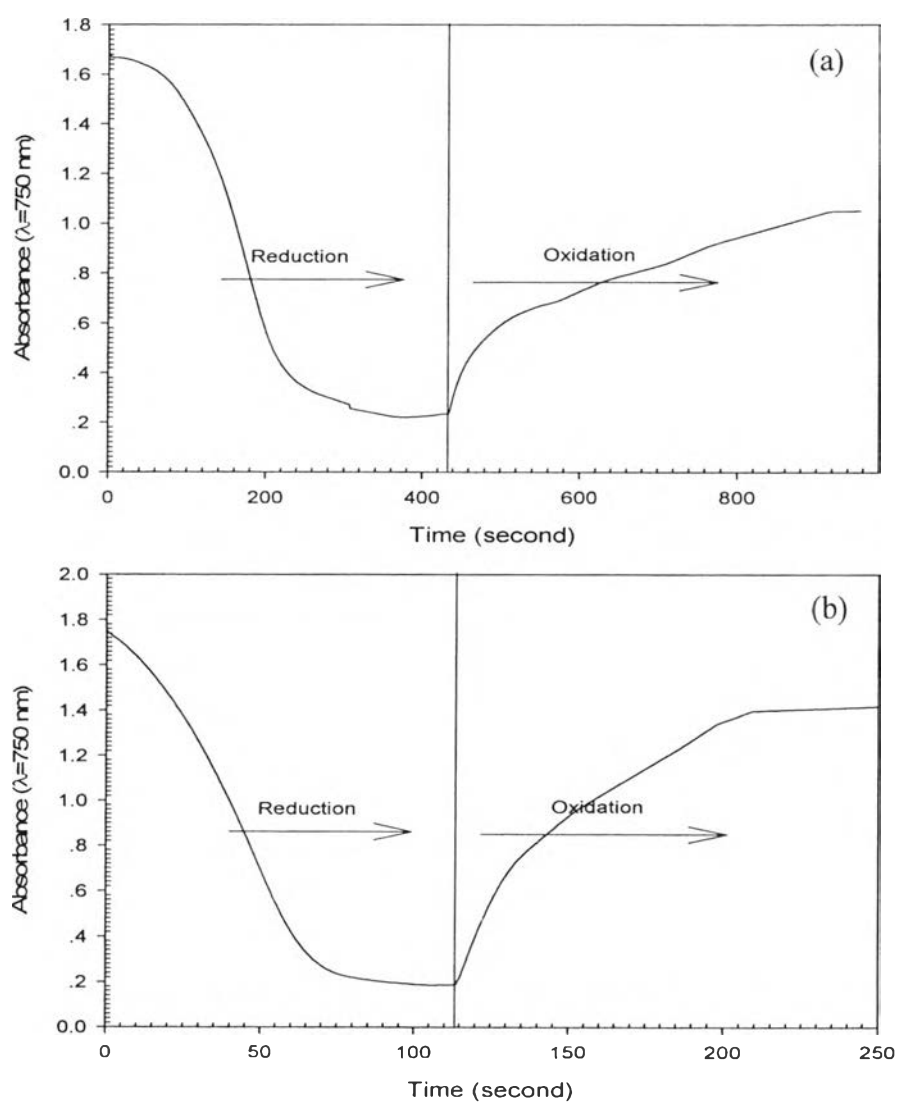
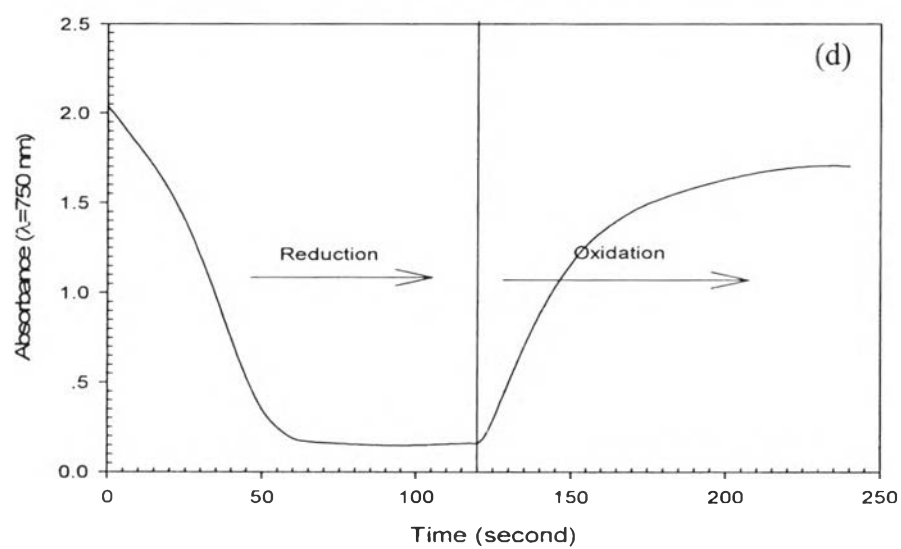
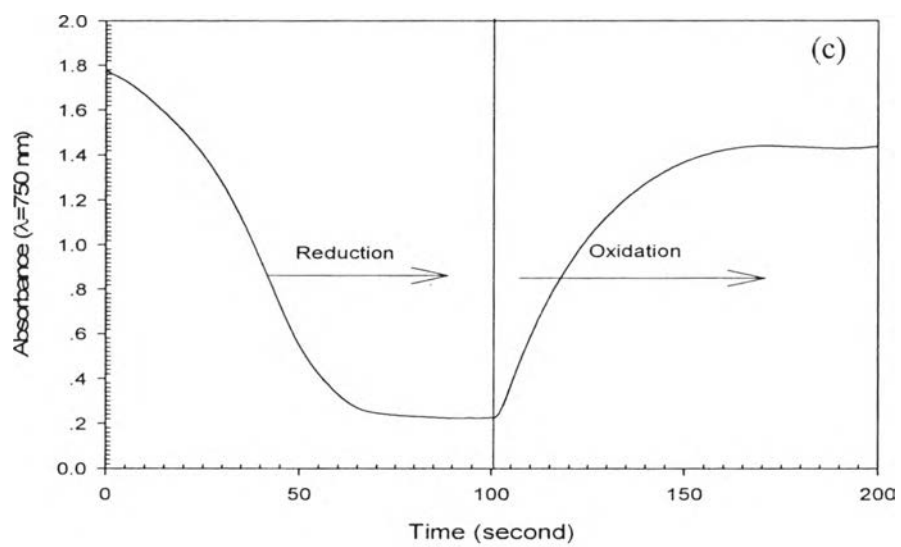


Figure E35 The effect of sulfuric concentration on the response time of PDMA synthesized in oxalic acid at 3 minutes.

The films of PDMA synthesized in nitric acid for 3 minutes were tested at 3.5 V in various concentration of sulfuric acid (10^{-6} , 10^{-5} , 10^{-4} , 10^{-3} , 10^{-2} , and 10^{-1} M). The absorption curves are shown in figures E36 (a) to (f), respectively.





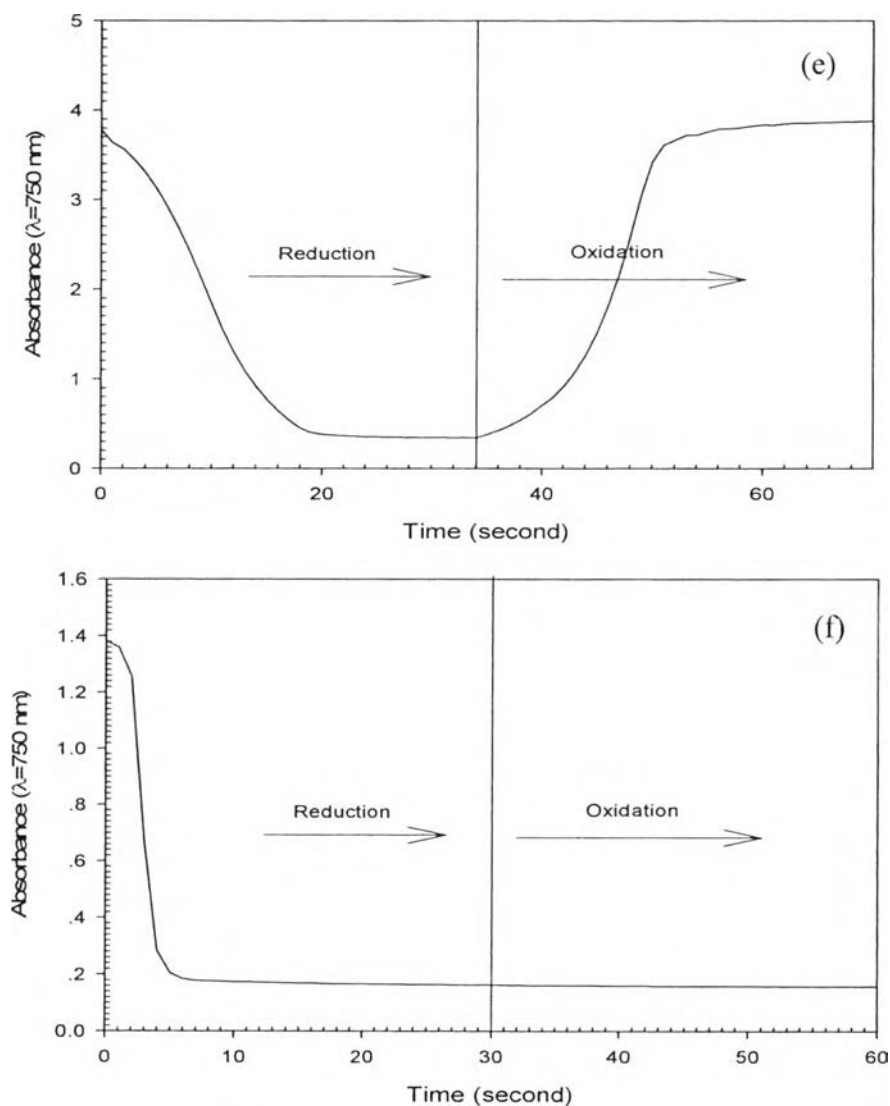


Figure E36 The absorption curves of PDMA synthesized in nitric acid for 3 minutes tested at 3.5 V (a) 10^{-6} , (b) 10^{-5} , (c) 10^{-4} , (d) 10^{-3} , (e) 10^{-2} , and (f) 10^{-1} M sulfuric acid.

The effect of sulfuric concentration on the reduction, oxidation, and response times of PDMA synthesized in nitric acid at 3 minutes is shown in Figures E37 to E39, respectively.

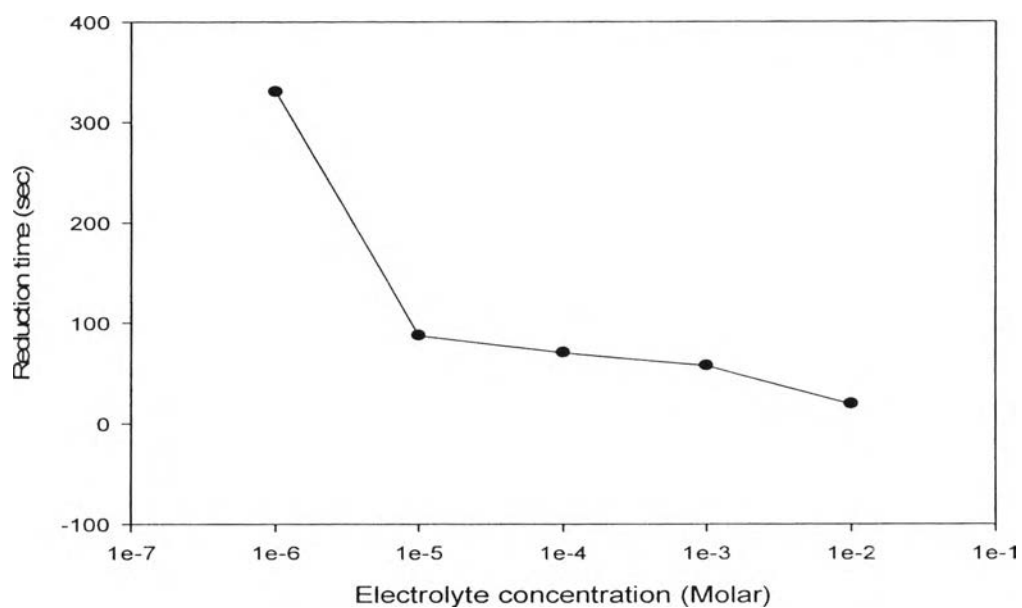


Figure E37 The effect of sulfuric concentration on the reduction time of PDMA synthesized in nitric acid at 3 minutes.

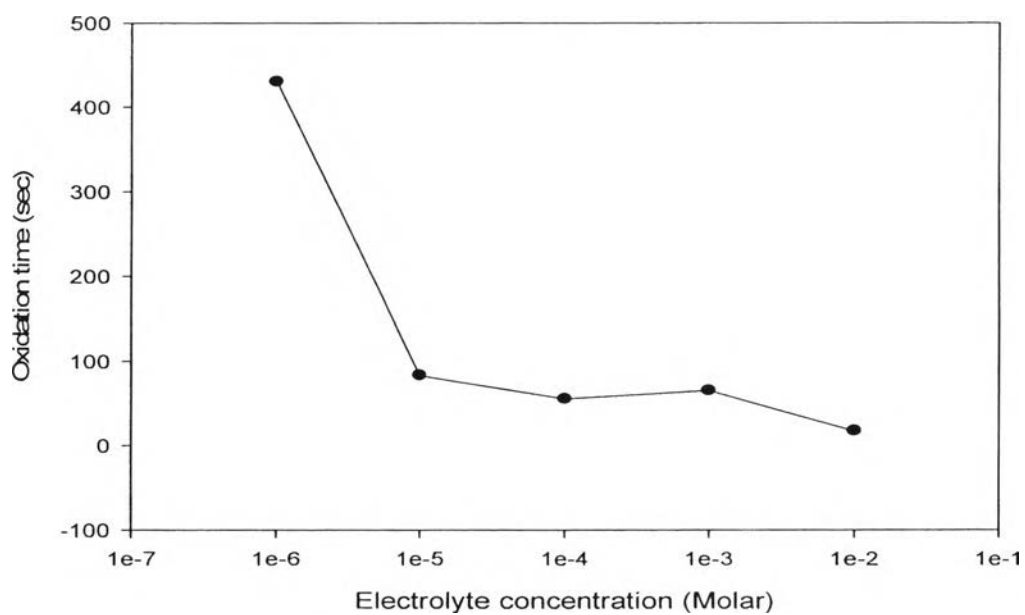


Figure E38 The effect of sulfuric concentration on the oxidation time of PDMA synthesized in nitric acid at 3 minutes.

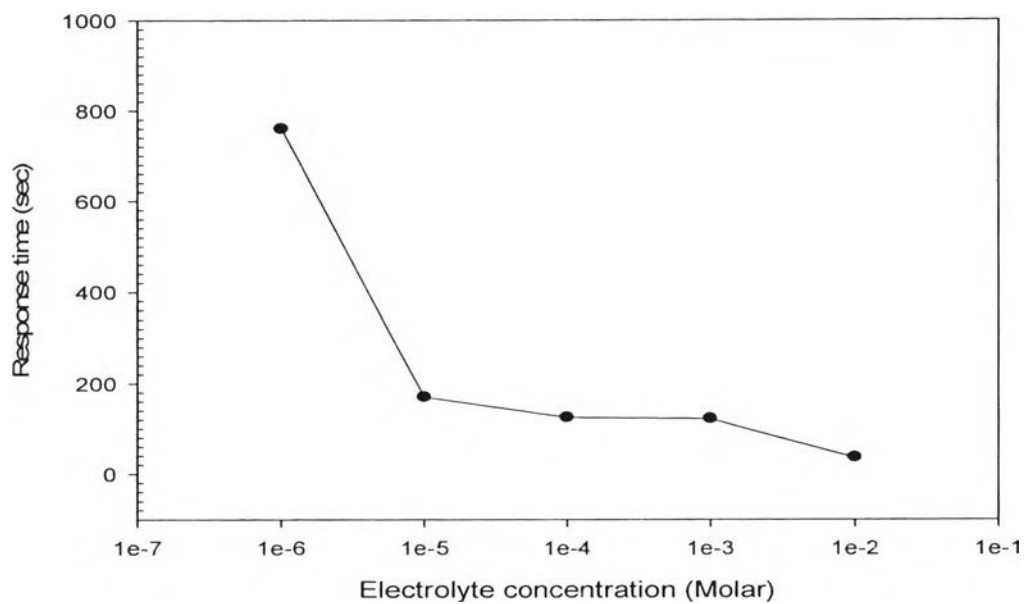
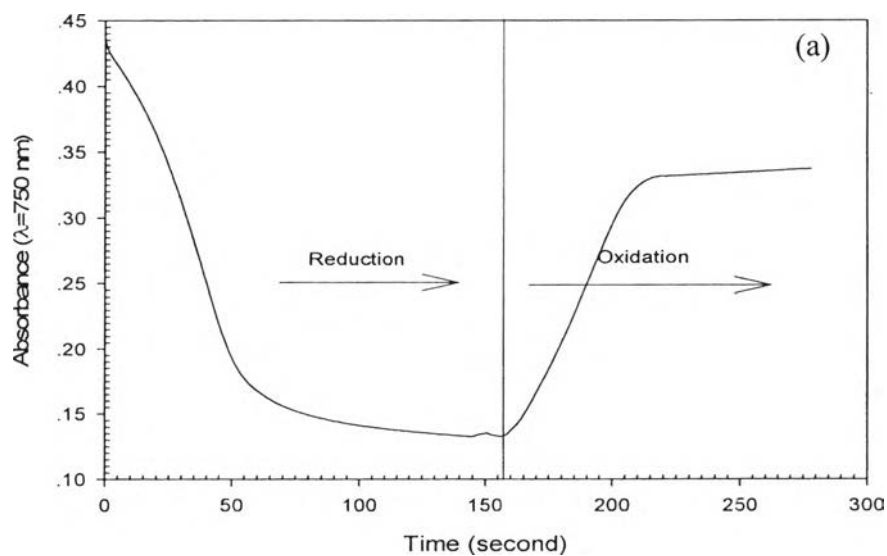
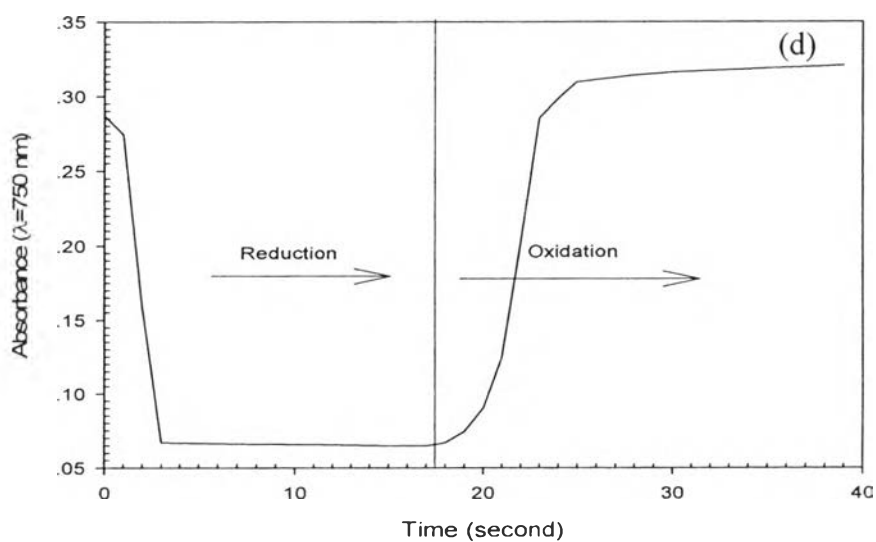
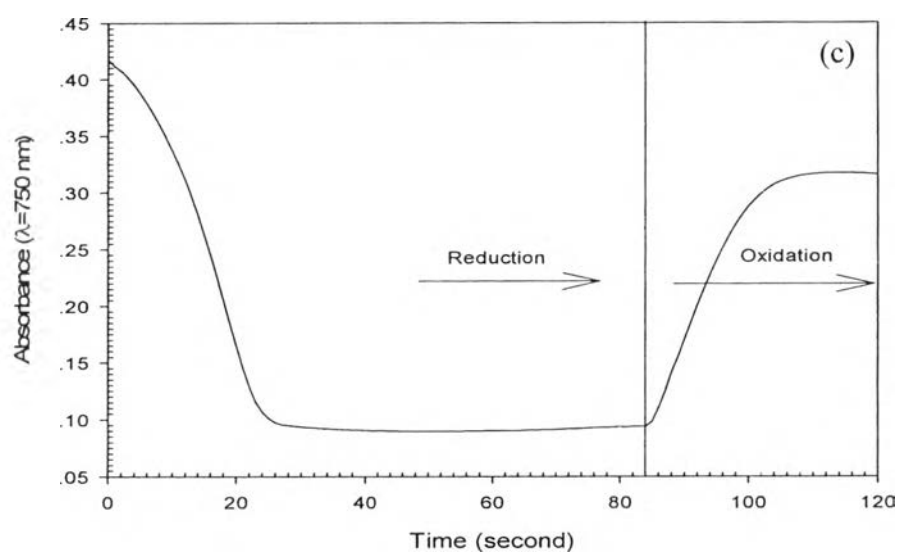
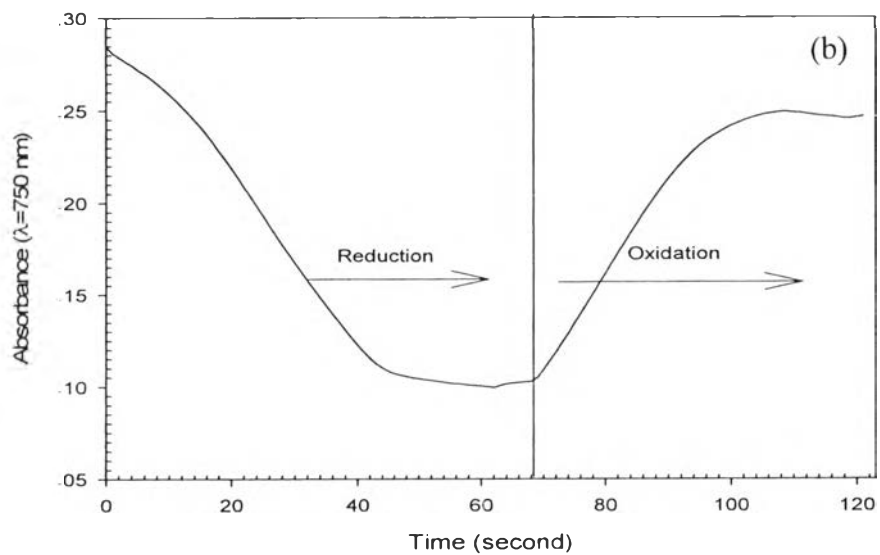


Figure E39 The effect of sulfuric concentration on the response time of PDMA synthesized in nitric acid at 3 minutes.

The films of PDMA synthesized in hydrochloric acid for 6 minutes were tested at 3.5 V in various concentration of sulfuric acid (10^{-6} , 10^{-5} , 10^{-4} , 10^{-3} , 10^{-2} , and 10^{-1} M). The absorption curves are shown in figures E40 (a) to (f), respectively.





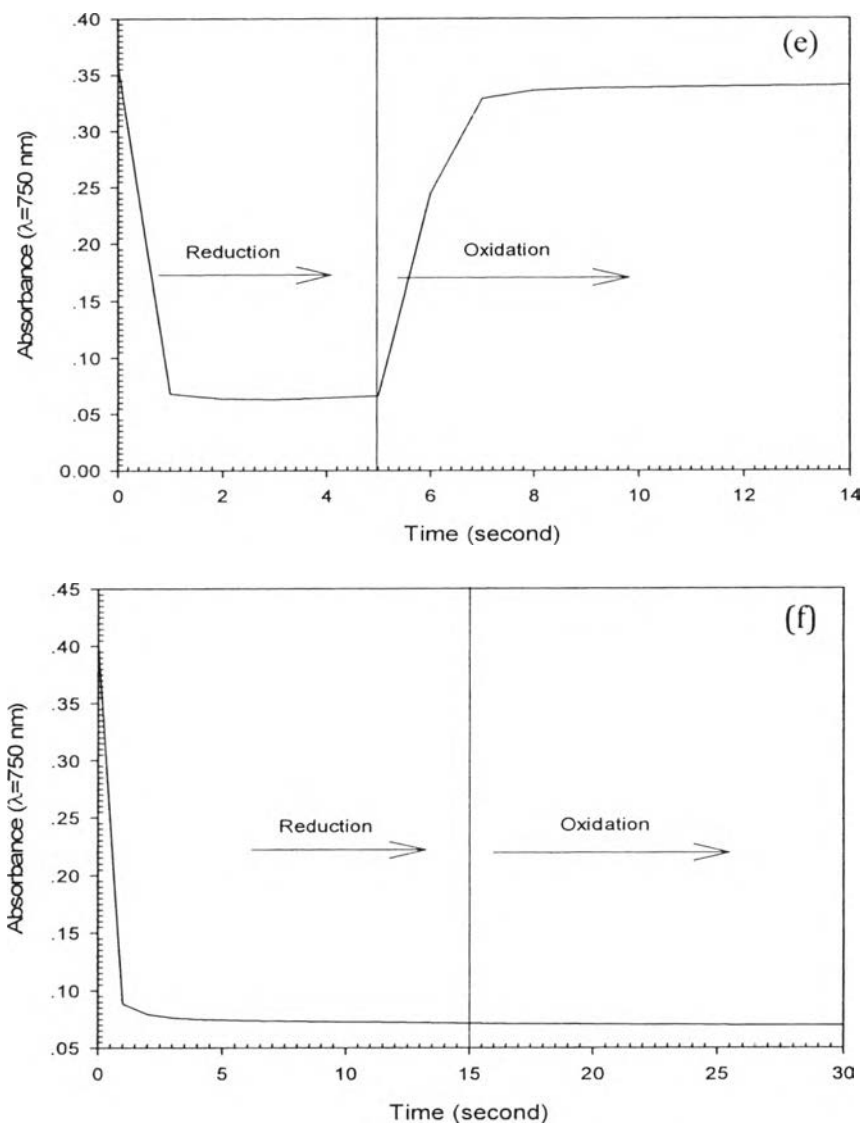


Figure E40 The absorption curves of PDMA synthesized in hydrochloric acid for 6 minutes tested at 3.5 V in (a) 10^{-6} , (b) 10^{-5} , (c) 10^{-4} , (d) 10^{-3} , (e) 10^{-2} , and (f) 10^{-1} M sulfuric acid.

The effect of sulfuric concentration on the reduction, oxidation, and response times of PDMA synthesized in hydrochloric acid at 6 minutes are shown in Figures E41 to E43, respectively.

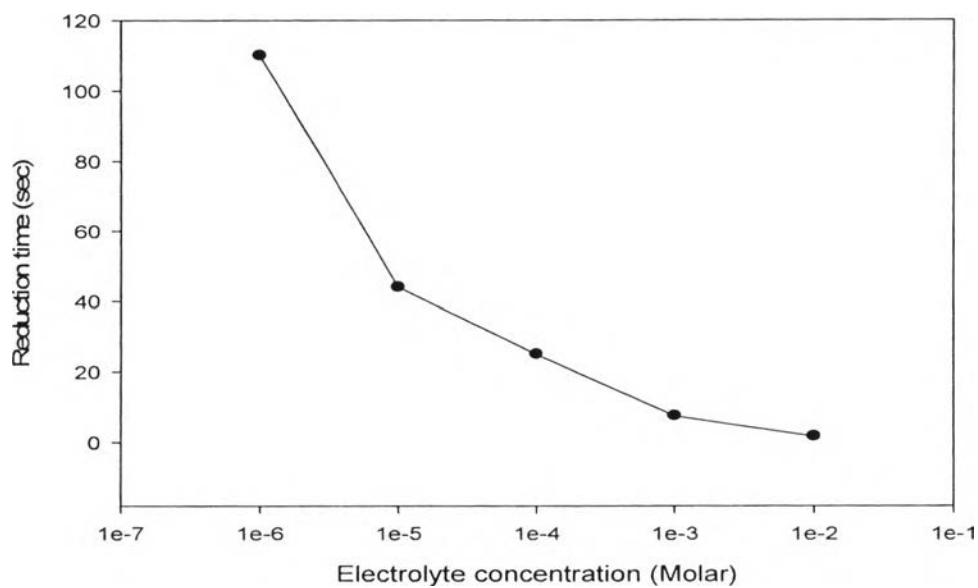


Figure E41 The effect of sulfuric concentration on the reduction time of PDMA synthesized in hydrochloric acid at 6 minutes.

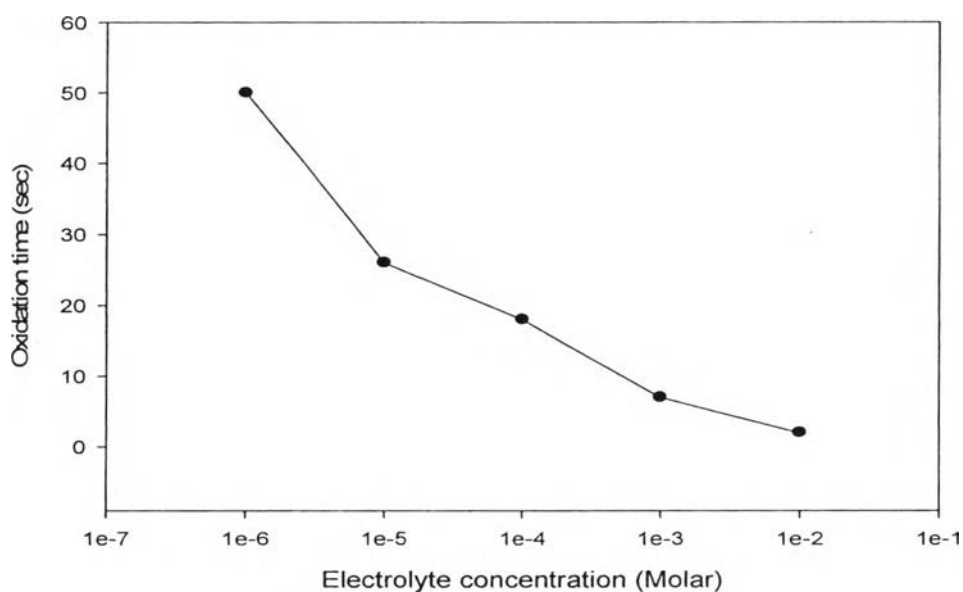


Figure E42 The effect of sulfuric concentration on the oxidation time of PDMA synthesized in hydrochloric acid at 6 minutes.

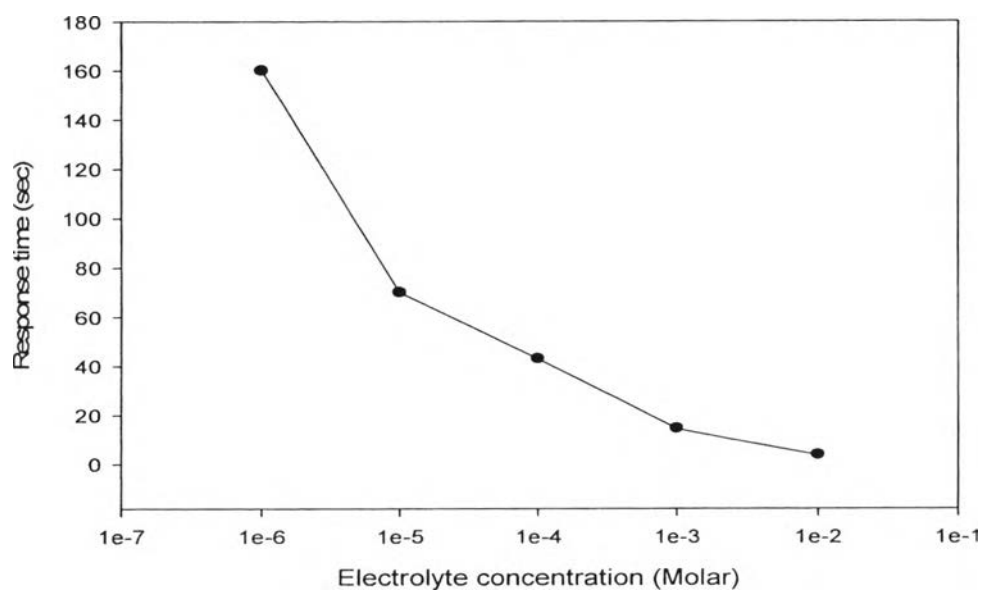


Figure E43 The effect of sulfuric concentration on the response time of PDMA synthesized in hydrochloric acid at 6 minutes.

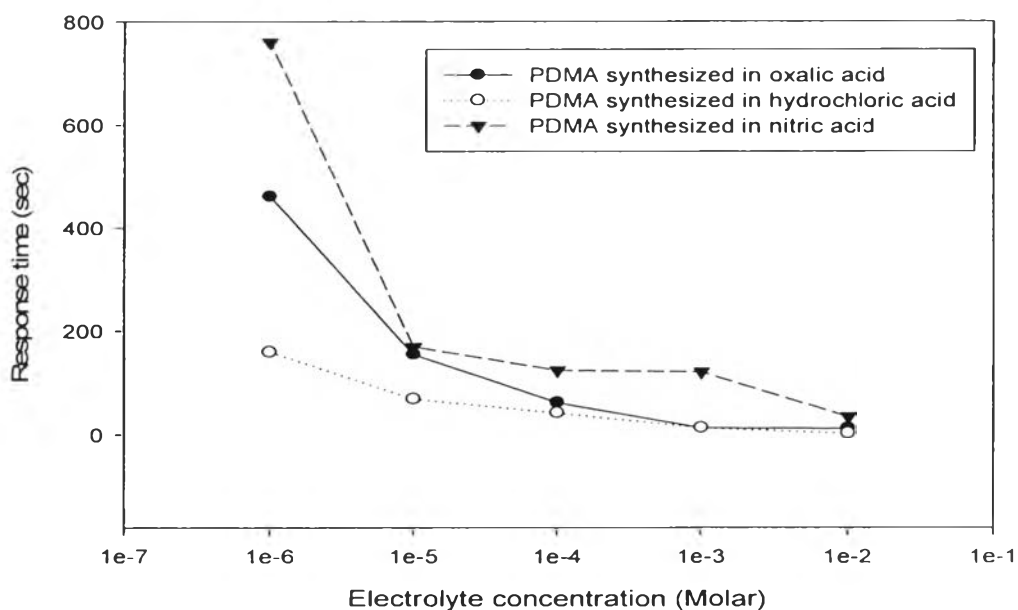


Figure E44 The effect of H_2SO_4 concentration on the response time at 3.5 V of PDMA coated ITO electropolymerized in each acid.

The optical contrast (ΔAbs) of PDMA/ITO was determined as the absorbance difference between the steady state value of the reduction and oxidation states as shown in Figure 45.

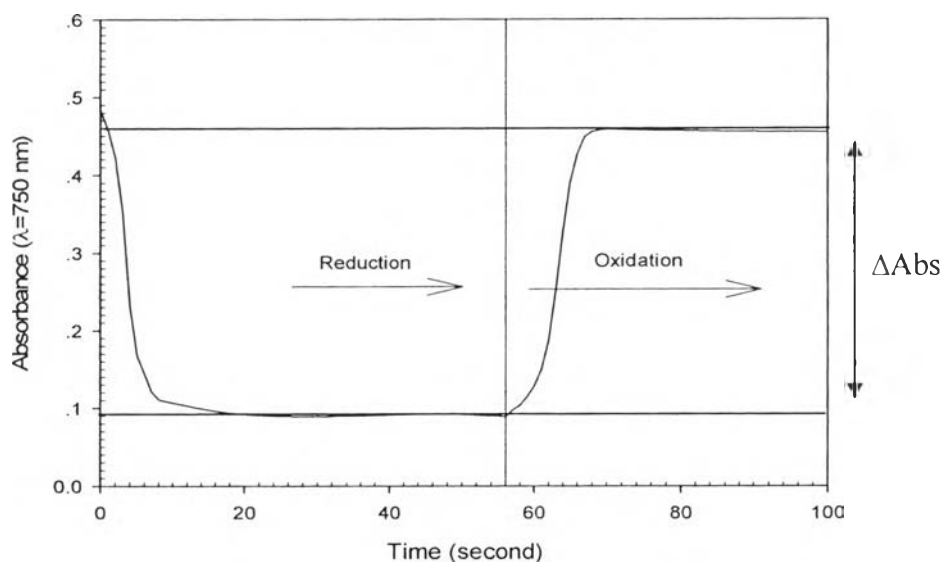


Figure E45 The optical contrast (ΔAbs) determination of PDMA synthesized in hydrochloric acid at 6 minutes with testing potential 2.0 V. via UV-Visible spectrophotometer.

Table E17 The optical contrast of PDMA at 6 and 10 minutes synthesized in **nitric** acid with applied potentials of 1.0, 1.5, 2.0, 2.5, 3.0, and 3.5 V

Applied potentials (V)	Absorbance (a.u.)				Optical contrast (ΔAbs)	
	6 min		10 min		6 min	10 min
	Reduction	Oxidation	Reduction	Oxidation		
1	0.7	2.37	0.5	2.7	1.67	2.2
1.5	0.65	3.4	0.6	3	2.75	2.4
2.0	0.125	2.6	0.5	3	2.475	2.5
2.5	0.35	2.6	0.4	2.9	2.25	2.5
3.0	0.25	2.518	0.12	1.9	2.268	1.78
3.5	0.25	2.4	0.35	2.95	2.15	2.6
				Average	2.26±0.36	2.33±0.30

Table E18 The optical contrast of PDMA at 6 and 10 minutes synthesis in **oxalic acid** with applied potentials of 1.0, 1.5, 2.0, 2.5, 3.0, and 3.5 V

Applied potentials (V)	Absorbance (a.u.)				Optical contrast (Δ Abs)	
	6 min		10 min		6 min	10 min
	Reduction	Oxidation	Reduction	Oxidation		
1	0.17	1.6	0.21	2.16	1.43	1.95
1.5	0.2	1.37	0.25	2.1	1.53	1.85
2.0	0.05	2.05	0.25	2.23	2.00	1.98
2.5	0.15	2.07	0.1	2.05	1.92	1.95
3.0	0.11	2.1	0.11	2.37	1.99	2.26
3.5	0.12	1.85	0.13	2.2	1.73	2.07
Average					1.77 \pm 0.24	2.01 \pm 0.14

Table E19 The optical contrast of PDMA synthesized for 6 and 10 minutes in **hydrochloric acid** with applied potentials of 1.0, 1.5, 2.0, 2.5, 3.0, and 3.5 V

Applied potentials (V)	Absorbance (a.u.)				Optical contrast (Δ Abs)	
	6 min		10 min		6 min	10 min
	Reduction	Oxidation	Reduction	Oxidation		
1	0.075	0.325	0.08	1.12	0.250	1.04
1.5	0.09	0.525	0.12	0.79	0.435	0.67
2.0	0.09	0.460	0.10	1.16	0.370	1.06
2.5	0.062	0.572	0.15	2.12	0.392	1.97
3.0	0.062	0.572	0.09	1.36	0.510	1.27
3.5	0.061	0.337	0.17	1.15	0.276	0.98
Average					0.37 \pm 0.097	1.17 \pm 0.43

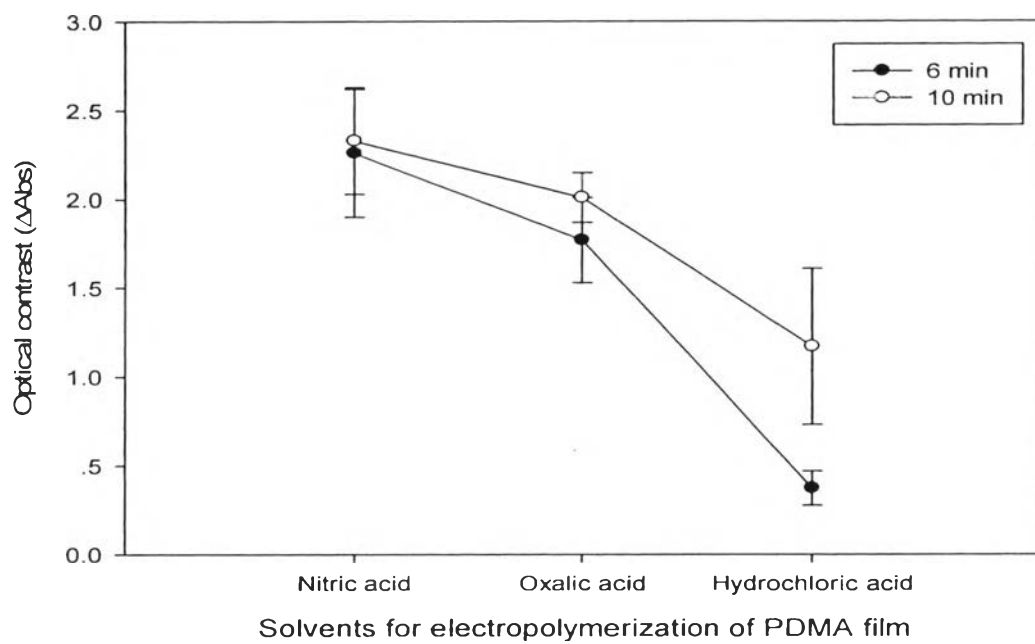


Figure E46 The optical contrast of PDMA coated ITO plastic via nitric, oxalic and hydrochloric acids for 6 and 10 minutes.

Appendix F Cyclic voltammetry (CV) of PDMA

Cyclic voltammetry (CV) is a widely used as an electroanalytical technique. It has wide applications in the study of redox processes, electrochemical properties of analytes in solution, and for understanding reaction intermediates (Bard & Faulkner, 2001).

The potential is usually measured between the reference electrode and the working electrode and the current is measured between the working electrode and the auxiliary electrode, also known as the counter electrode. This data is then plotted as current versus potential as shown in Figure F1.

The forward scan produces a current peak for any analytes that can be reduced (or oxidized depending on the initial scan direction) through the range of the potential scanned. The current increases as the potential reaches the reduction potential of the analyte, but then decreases as the concentration of the analyte is depleted close to the electrode surface. (Songa, 2008)

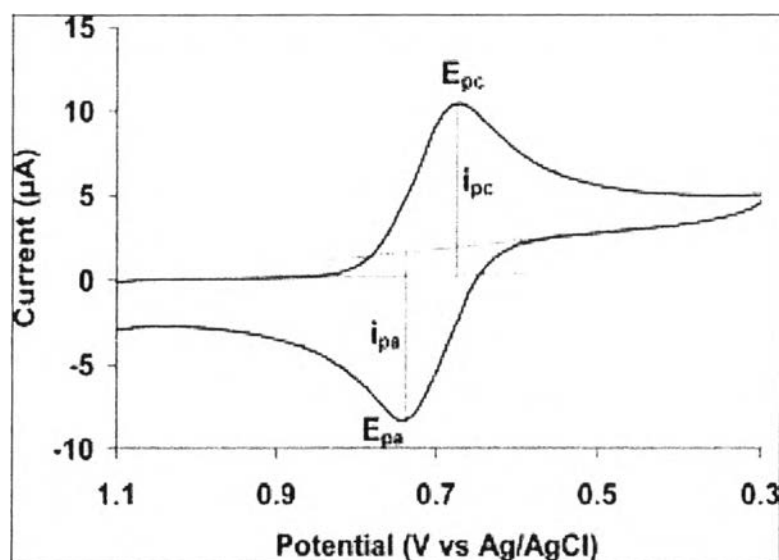


Figure F1 A typical cyclic voltammogram.

If the redox couple is reversible, then reversing the applied potential re-oxidizes the product formed in the first reduction reaction, thus producing a reverse

current opposite to the forward scan. The important parameters in a cyclic voltammogram are the peak potentials (E_{pc} , E_{pa}) and peak currents (I_{pc} , I_{pa}) of the cathodic and anodic peaks, respectively.

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 H_2SO_4 background electrolyte, assembled with a three electrodes cell consisting of a saturated calomel electrode (SCE) as a reference electrode, a 5 cm length stainless steel wire as a counter electrode and 2 cm^2 PDMA/ ITO plastic was used as the working electrode, as shown in Figure F2.

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 cm^2 . The electrochromic device was compiled with the following configuration:

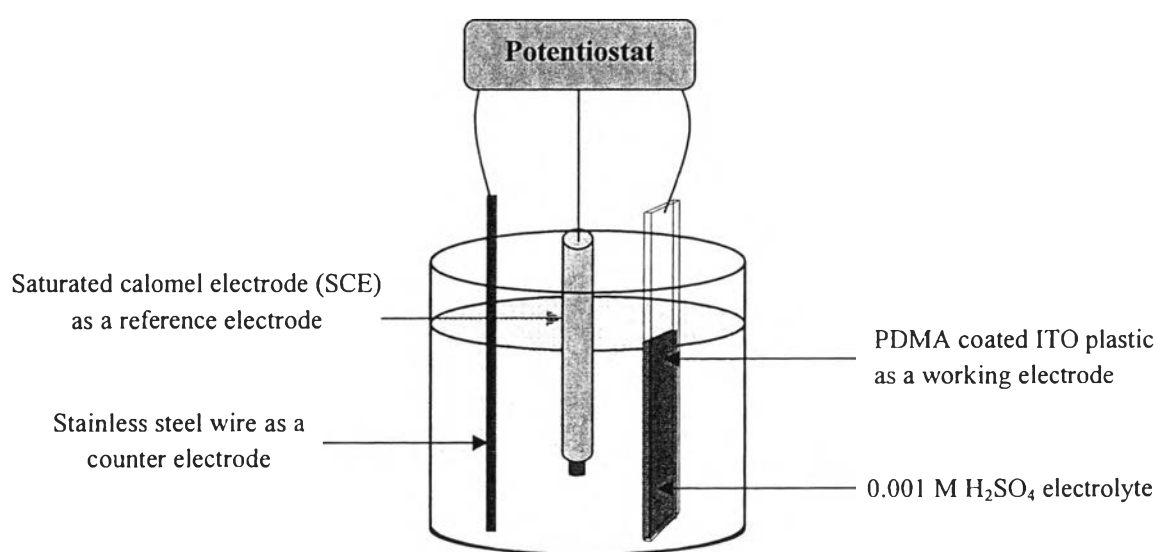


Figure F2 Schematic of a electrochemical cell of PDMA/ITO for the investigation of the redox properties via cyclic voltammetry.

Cyclic voltammograms (CV) of PDMA/ITO synthesized in oxalic, nitric and hydrochloric acids at various times are shown in Figures F3 to F5, respectively.

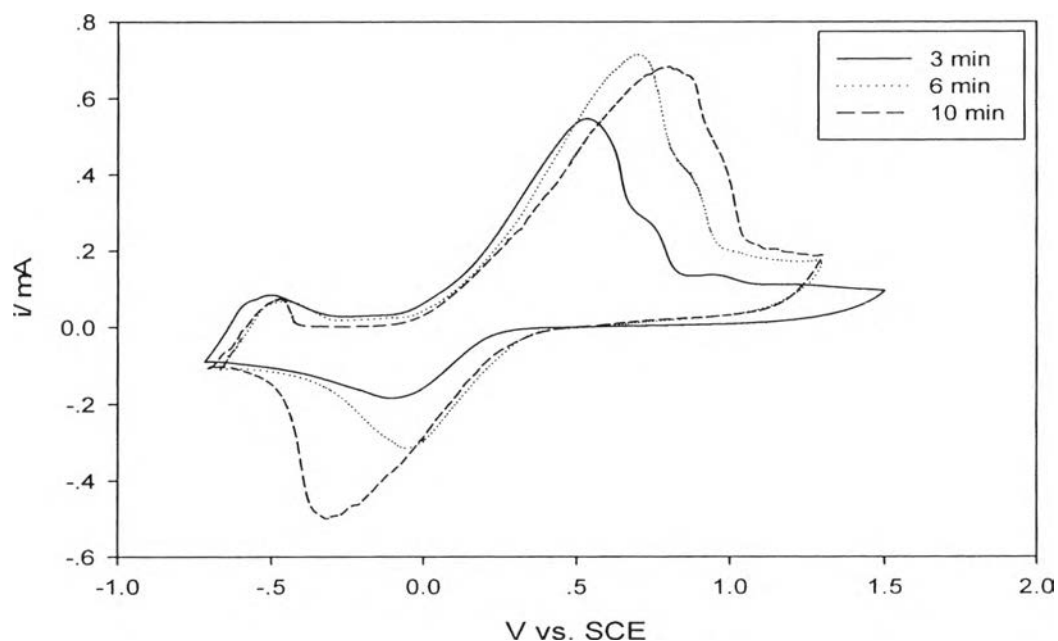


Figure F3 Cyclic voltammograms of PDMA/ITO synthesized in oxalic acid at 3, 6, and 10 minutes.

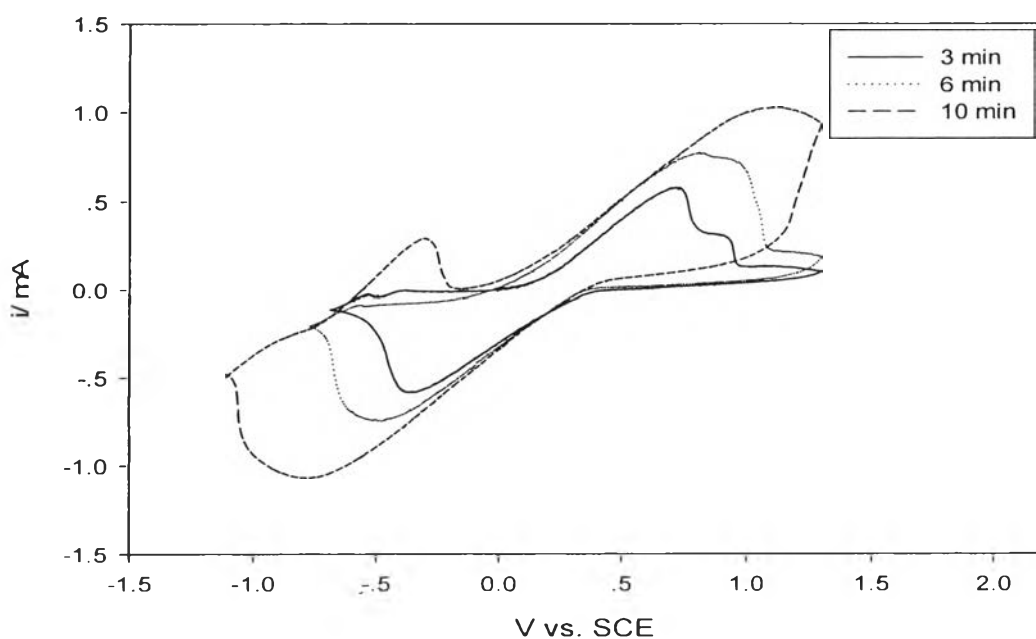


Figure F4 Cyclic voltammograms of PDMA/ITO synthesized in nitric acid at 3, 6, and 10 minutes.

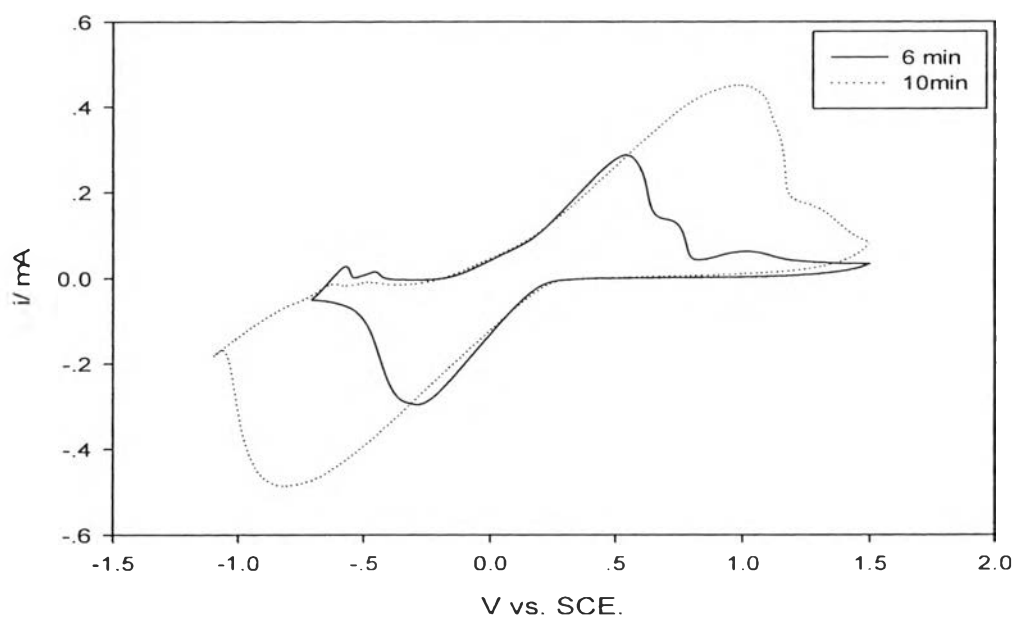


Figure F5 Cyclic voltammograms of PDMA/ITO synthesized in hydrochloric acid at 6 and 10 minutes.

The PDMA deposited ITO via all acids shows two main redox processes in which the first weak indicates the oxidation at around -0.5 V related to the leucocmeraldine/ emeraldine transformation, and the second sharp peak corresponds to the emeraldine/ pernigraniline transformation (Gazotti *et al.*, 1997).

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 F1.

Table F1 The peak potentials (E_{pc} , E_{pa}) and peak currents (I_{pc} , I_{pa}) of the PDMA synthesized in oxalic, nitric, and hydrochloric acids

Acids	Polymerization time (minutes)	Current (mA)		Potential (V)	
		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 acid	6	0.3	0.3	-0.3	0.5
	10	4.7	4.7	-0.82	0.98

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.

Appendix G The Morphology and Thickness of PDMA

The field-emission scanning electron microscopy (HITACHI S4800) is 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 G1.

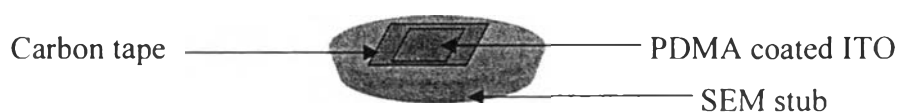
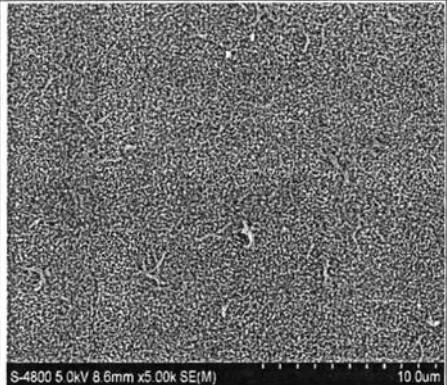
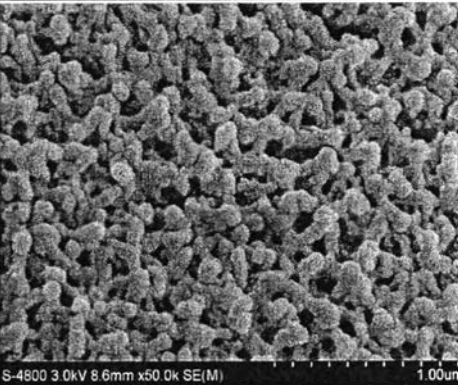
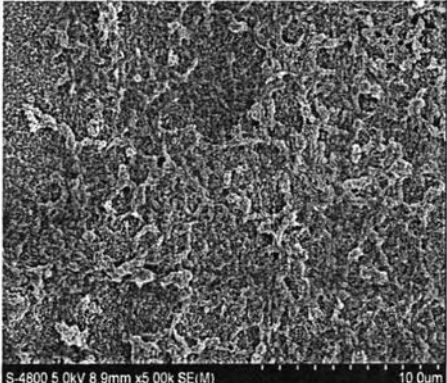
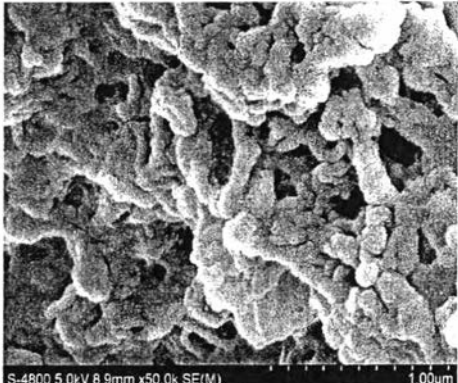


Figure G1 The schematic for the investigation of PDMA surface morphology via FE-SEM.

The FE-SEM images of PDMA surface electrodeposited at 3 minutes via oxalic and nitric acids are illustrated in Table G1.

Table G1 Surface Morphology of PDMA polymerized for 3 minutes via various acids

Acids	SEM Images	
	5.00k	50.0k
Oxalic acid		
Nitric acid		

The FE-SEM images of PDMA surface electrodeposited via oxalic, nitric, and hydrochloric acids for 6 and 10 minutes are illustrated in Tables G2 and G3, respectively.

Table G2 Surface Morphology of PDMA polymerized for 6 minutes via various acids

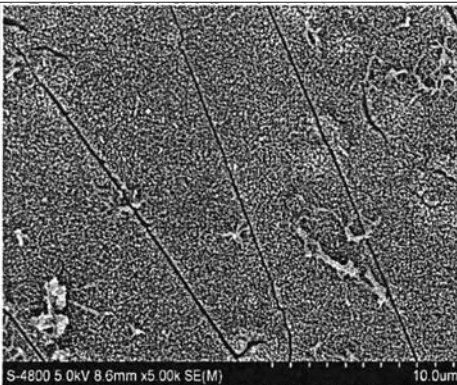
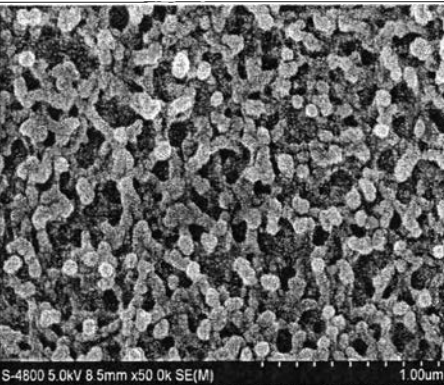
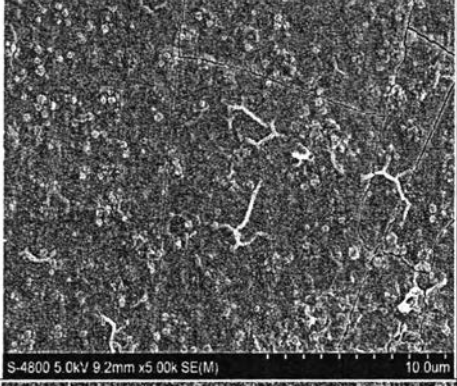
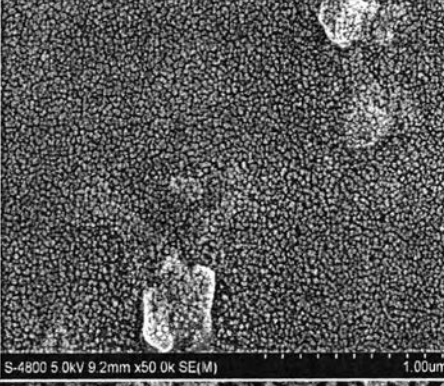
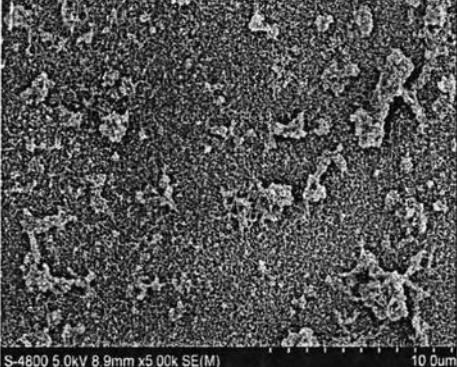
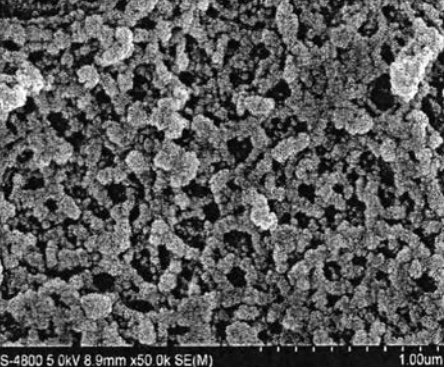

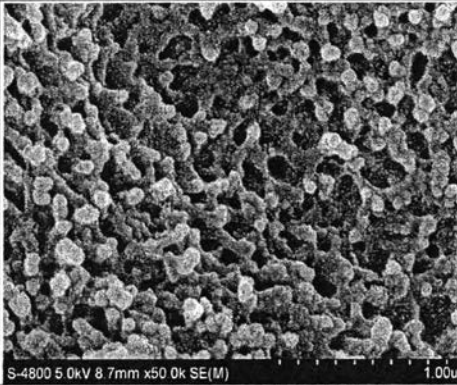


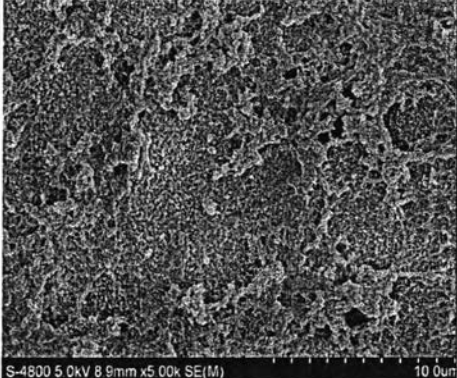
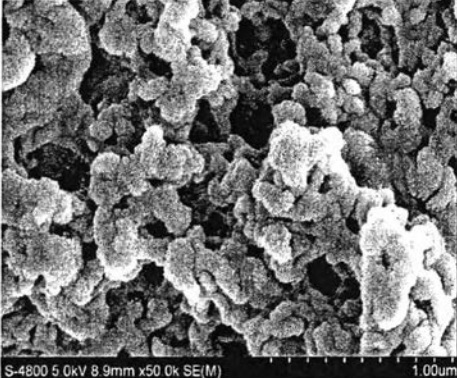
Acids	SEM Images	
	5.00k	50.0k
Oxalic acid	 S-4800 5.0kV 8.6mm x5.00k SE(M) 10.0um	 S-4800 5.0kV 8.5mm x50.0k SE(M) 1.00um
Hydrochloric acid	 S-4800 5.0kV 9.2mm x5.00k SE(M) 10.0um	 S-4800 5.0kV 9.2mm x50.0k SE(M) 1.00um
Nitric acid	 S-4800 5.0kV 8.9mm x5.00k SE(M) 10.0um	 S-4800 5.0kV 8.9mm x50.0k SE(M) 1.00um

Table G3 Surface Morphology of PDMA polymerized for 10 minutes via various acids

Acids	SEM Images	
	5.00k	50.0k
Oxalic acid		
Hydrochloric acid		
Nitric acid		

The morphology of PDMA is different when PDMA was synthesized in the different acids indicating that the type of acids plays a role in directing the structure and morphology and aligning the monomers. PDMA from oxalic acid shows high porosity microfiber structure 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 and with particle aggregation.

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.

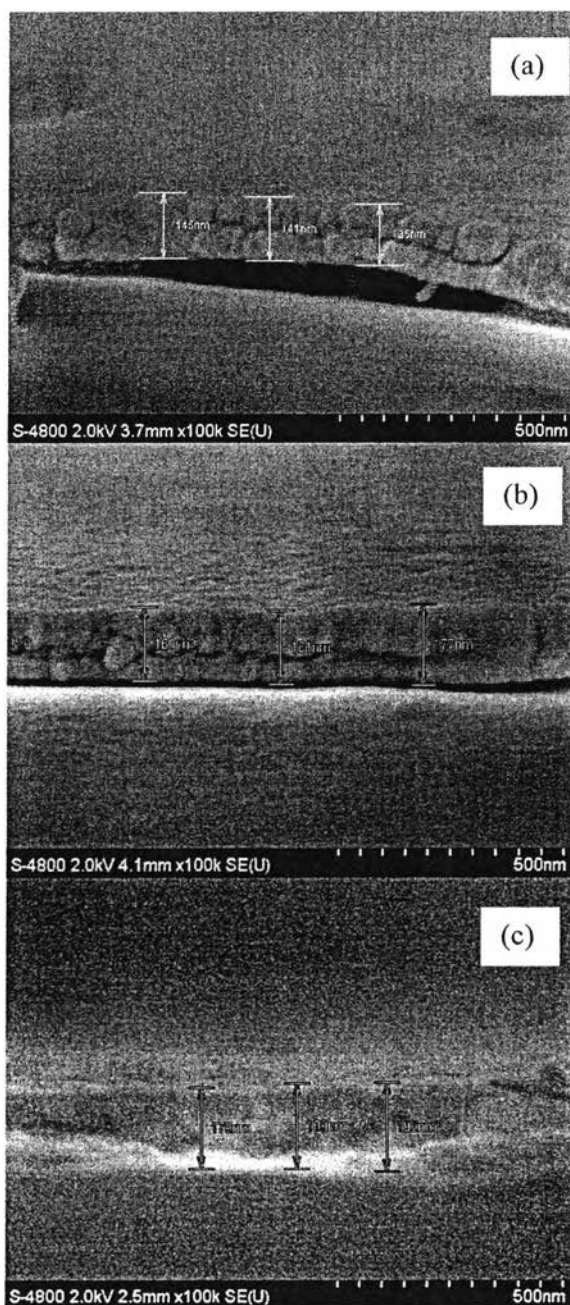


Figure G2 Cross-sectional FE-SEM of PDMA/ITO synthesized in nitric acid at (a) 3 min, (b) 6 min, and (c) 10 min.

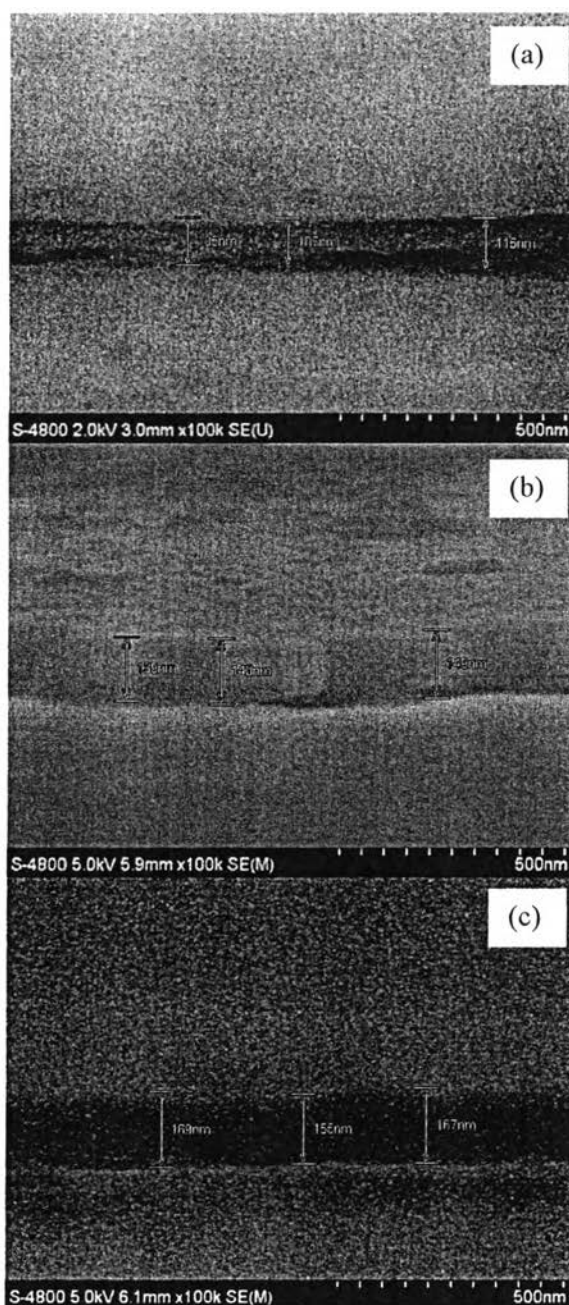


Figure G3 Cross-sectional FE-SEM of PDMA/ITO synthesized in oxalic acid at (a) 3 min, (b) 6 min, and (c) 10 min.

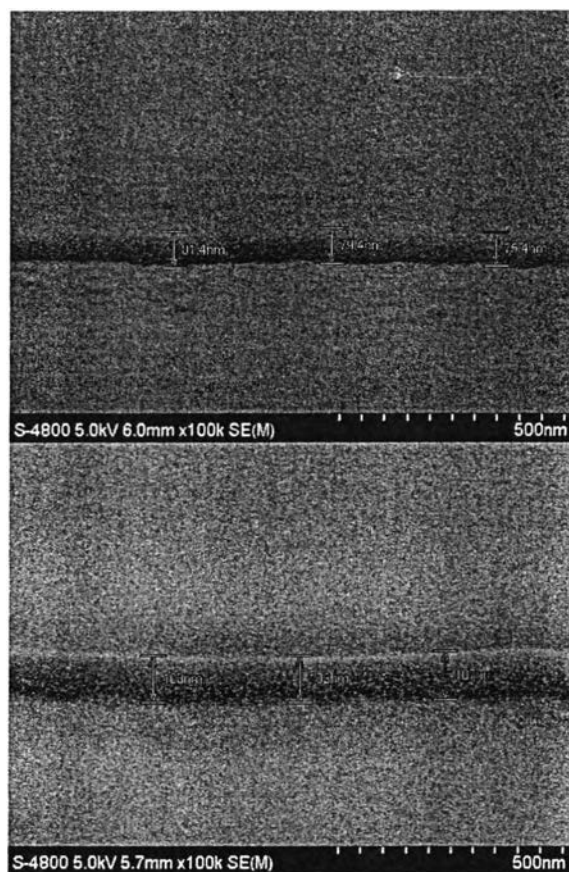


Figure G4 Cross-sectional FE-SEM of PDMA/ITO synthesized in hydrochloric acid at (a) 6 min and (b) 10 min.

Table G4 Thickness of PDMA/ITO synthesized in various acids at 3, 6, and 10 minutes dipping time

Acids	Thickness (nm)		
	3 min	6 min	10 min
Oxalic acid	109±5	144±5	164±8
Nitric acid	140±5	166±9	188±8
Hydrochloric acid	-	78±3	104±2

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. 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 G4. 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.

CURRICULUM VITAE

Name: Ms. Bureerat Suephatthima

Date of Birth: March 25, 1988

Nationality: Thai

University Education:

2005-2010 Bachelor Degree of chemistry, Faculty of Science, Mahidol University, Bangkok, Thailand

Proceedings:

1. Suephatthima, B. and Sirivat A. (2012, April 24). Physical and Electro - chromic Properties of Poly (2,5-dimethoxy Aniline) Synthesized in Oxalic, Nitric, and Hydrochloric Acids. Proceedings of the 3rd Research Symposium on Petrochemical, and Materials Technology and the 18th PPC Symposium on Petroleum, Petrochemicals, and Polymers, Queen Sirikit National Conventional Center, Bangkok, Thailand.

Presentations:

1. Suephatthima, B; and Smith, S.M. (2010, January 21- 23) Chemical and Physical properties of Fe-doped ZnO nanorods. Paper presented at Pure and Applied Chemistry International Conference (PACCON 2010), Ubon Ratchathani, Thailand.
2. Suephatthima, B. and Sirivat A. (2012, March 25-29) Electrochromic Properties of Poly (2,5 dimethoxy aniline) Synthesized in Various Acids. Paper presented at 243rd American Chemical Society National Meeting, San Diego, California, USA.
3. Suephatthima, B. and Sirivat A. (2012, April 24). Physical and Electro - chromic Properties of Poly (2,5-dimethoxy Aniline) Synthesized in Oxalic, Nitric, and Hydrochloric Acids. Paper presented at the 3rd Research Symposium on Petrochemical, and Materials Technology and the 18th PPC Symposium on Petroleum, Petrochemicals, and Polymers, Queen Sirikit National Conventional Center, Bangkok, Thailand.

