Result of Scan Rate Variation on Electrochemical polymerization of OPD (Ortho Phenylene Diammine) coated PANI and PPD (Para Phenylene Diammine) coated PANI

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Abstract- OPD or PPD itself is not showing electrochemical activity when coated/polymerized on bare platinum plate.As shown by many researches the mixture of PPD and OPD and Aniline monomers may shows the electrochemical activity but here we used PANI film coated on platinum as an electrode or a catalyst to make PPD or OPD as electrochemically active. The increasing concentration of OPD and PPD monomer with variation of scan rate and hence the Charge storage capacity of polymer films are studied here by using the cyclic voltammetry technique.

The decrease in the charge storage capacity and increase in peak current of the above different polymeric films is found as increasing with the increase in the monomer concentration. When the scan rate varied from high to low. The UV-visiblespectra for the above polymers with their monomer solutions is measured and discussed.

Key words: Para phenylene diammine, Ortho phenylene diammine, Peak current, Scan rate etc.

Introduction:

Cyclic voltammetry is one of the best tool to study the ICP's. The electrochemically prepared polymers/ICP's/Films are further characterized and applied for various purposes. During electrochemical polymerization of different monomers a variety of studies can be carried out to obtained the desired products. A lot chemical reactions for their oxidation and reduction are carried out electrochemically and the prepared polymers are used in the variety of fields like corrosion studies, batteries, catalysis and electrochromic devices etc.

During the electrochemical preparation of polymer the scan rate of the working electrode is plays an important role. We can say that the Scan rate is nothing but the potential range applied per unit time to the electrode. The current generated during the electrochemical polymerization is how dependent on the scan rate can be accounted and the various different aspects of the electrochemical reaction can be explained on the basis of this data.

Here we prepare the pPPD (poly para phenylene diamine) and pOPD (poly ortho phenylene diamine) polymers by using cyclic voltammetry method and the effect of scan rate during the formation of these polymers is studied in detailed. Lida khalifi et al do the simulation for the scan rate effect and its mathematical concept.

These monomers OPD and PPD are not polymerized on the bare platinum plate so to make them electrochemically active many researchers used either the mixture of these with each other or with other monomer.

Here we achieve the electrochemical activity of PPD and OPD by coating them on the platinum plate on which already the PANI (poly-aniline) is coated/polymerized. The PANI (poly-aniline) is acts as the catalyst or a reagent for electrochemical polymerization of PPD and OPD. The different concentrations of these monomers are further used to polymerize them at different scan rate and then how the scan rate influenced the peak current and the charge stored during their preparation is explained here.

The relation between the peak current generated during the formation of the above polymers and the scan rate is explained with the relation of Randles-sevcik equation. With the help of the Randles-Sevcik plots the conclusions are discussed here.

Result and Discussion:

The Aniline Merck make used after time to time distillation in Rotavapp, PPD and OPD both are otto Kemi make and are used after re-crystallization in Ethyl alcohol (China make,100 percent pure ethanol). The CHI6089D potentiostat and Pine Rotating Disc Electrode set-up is used to polymerize the above chemicals by cyclic voltametrically. The three electrodes system setup is used Platinum as RDE working electrode, the SCE(Saturated Calomel Electrode) as the reference electrode while the platinum wire gauze as the auxiliary electrode.

All required concentrations of OPD (1.25,2.50,5,10.0,20.0 millimolar (mM) and PPD(1.25, 2.50, 5,10, 20 millimolar,mM) are prepared in 0.5M H₂SO₄ (98% concentrated, Merck make) solvent/dopant which is prepared in triple distilled water.

The aniline is polymerized on platinum RDE by cyclic voltmetrically at a Scan Rate of 50 Volts/Second with 3000 RPM speed. The 98 cycles are kept constant every time to get the constant current (0.7Ampere) film or base. The monomers PPD and OPD are polymerized on this polyaniline film for every different concentration of OPD and PPD for their scan rate study.

The cyclic voltametric peak currents obtained from software for every concentration used of OPD and PPD is noted with the charge stored in every film. From the peak current (Ip) values the graphs are plotted for Scan rate and peak current, Sqrt scan rate and peak current as per Randles-Sevcik equation the conclusions are drawn. The graphs of Charge obtained during every polymeric film are plotted against the scan rate and peak current.

#1.25 mM OPD coated PANI:



Fig 1:	Current	Vs Voltage	Plots of 1.2	5 mM OPD	coated PANI	(5 Mv/Secon	ds to 10	0 Mv/Seconds)
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Sr. No.	Scan Rate (v) in Volts/Sec	$Sqrt$ $Scan$ $Rate$ $(v^{1/2)}$	Peak Potential (Ep)	Peak Current (Ip) in Ampere/cm ²	Log Ip	Charge (Q) in Film in Coulomb
1	5	2.24	0.139	0.0000667	-4.1756	0.025460
2	10	3.16	0.144	0.0001452	-3.8377	0.012030
3	25	5.00	0.149	0.0003644	-3.4385	0.004788
4	50	7.07	0.159	0.0006618	-3.1793	0.002443
5	100	10.0	0.168	0.001196	-2.9223	0.001318

Table 1: Scan Rate Study of 1.25 mM OPD coated PANI:

The Scan Rate study for 1.25 mM OPD coated PANI is done by scanning the electrode film from 5 Mv /seconds (Peak Crrent-6.672E-5 Ampere/Cm²) to 100 Mv/Seconds (Peak Crrent-1.196E-3 Ampere/Cm²) at constant speed of the RDE was 3000 RPM. It is found that the product peak is found at potential 0.139 volt to 0.168 volt no marginal changes are found

in the potential value but it changes from 0.139 volt (5 Mv/Seconds) to 0.168(100 Mv/Seconds) and peak currents are increased from 6.672E-5 Ampere/Cm²(at 5 Mv/Seconds) to 1.196E-3 Ampere/Cm² indicates the formation of radical OPD is more as more concentration of monomer used. Charge stored in the film is changed from 2.54E-2 Coulomb to 1.31E-3 coulomb. **#2.50 mM OPD coated PANI:**



Fig 2: Current Vs Voltage Plots of 2.50 mM OPD coated PANI at 5 Mv/Seconds to 100 Mv/Seconds.

The Scan Rate study for 2.5 mM OPD coated PANI is done by scanning the electrode film from 5 Mv/seconds (Peak Crrent-6.24E-4 Ampere/Cm²) to 100 Mv/Seconds (Peak Crrent-1.16E-3 Ampere/Cm²) at constant speed of the RDE was 3000 RPM. It is found that the product peak is found at potential 0.119 volt to 0.156 volt. Marginal changes are found in potential and peak currents are increased from 6.24E-4 Ampere/Cm²(at 5 Mv/Seconds) to 1.16E-3 Ampere/Cm² indicates the formation of radical OPD is more as more concentration of monomer used.

The relationship of Scan Rate with peak current, square root of peak current and charge in the film is plotted with respective to Randles-Sevcik equation. It is found that the charge in the film for 2.5 mM OPD coated PANI film changes from 2.94E-2 Coulomb to 9.31E-4 coulomb the decrease in the capacitive charge is the symbol of at less scan rate the more capacitive film is formed while as the Scan rate increases the capacitive charge is decreases in the film.

Sr.	Scan Rate	Sqrt Scan	Peak	Peak Current	log Ip	Charge stored
190.	(V) III (Volts/Sec)	Kate (v.	(Ep)Volts	(1p) InAmpere/Cm ²		in Coulomb
1	5	2.2361	0.119	0.0000624	-4.2047	0.0294800
2	10	3.1623	0.136	0.0001287	-3.8904	0.0115700
3	25	5.0000	0.149	0.0003287	-3.4832	0.0057660
4	50	7.0711	0.128	0.0005932	-3.2268	0.0029970
5	100	10.0000	0.156	0.0011620	-2.9348	0.0009319

Table 2: Scan Rate Study of 2.50 mM OPD coated PANI:

5.0 mM OPD coated PANI:



Fig	3:	Current	Vs	Voltage	Plots	of 5.0	mM Ol	PD coate	ed PAN	[at 5	Mv	/Secon	ds to	100	Mv/S	econds	3.
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Sr. No.	Scan Rate (v) in Volts/Sec	Sqrt Scan Rate $(\mathbf{v}^{1/2})$	Peak Potential (Ep) in volts	Peak Current (Ip) in Ampere/Cm ²	log Ip	Charge (Q) in film in Coulomb
1	5	2.2361	0.117	0.0000662	-4.1789	0.0725000
2	10	3.1623	0.14	0.0001252	-3.9024	0.0515000
3	25	5.0000	0.147	0.0003613	-3.4421	0.0196700
4	50	7.0711	0.156	0.0006626	-3.1787	0.0009728
5	100	10.0000	0.165	0.0013510	-2.8693	0.0004942

Table 3: Scan Rate Study of 5.0 mM OPD coated PANI:

For 5.0 mM OPD coated PANI as the Scan Rate increases from 5Mv/Seconds to 100 Mv/Seconds then the peak current is decreases from 6.62E-5 Ampere/Cm² to 1.35E-3 Ampere/Cm² and the charge stored in the film is changed from 7.25E-2 Coulomb to 4.94E-4 coulomb.





Sr. No.	Scan Rate (v) in (Volts/Sec)	Sqrt Scan Rate(v ^{1/2)}	Peak potential (Ep) in volts	Peak Current (Ip)In Ampere/Cm ²	Log IP	Charge (Q) in film in Coulomb
1	5	2.2360	0.114	0.0000607	-4.216811309	0.17600
2	10	1.0000	0.138	0.00015	-3.823908741	0.09300
3	25	5.0000	0.140	0.003997	-2.398265852	0.00456
4	50	7.0710	0.144	0.007821	-2.106737714	0.00218
5	100	10.0000	0.156	0.01614	-1.79209647	0.00120

Table 4: Scan Rate Study of 10 mM OPD coated PANI:

For 10 mM OPD coated PANI as the Scan Rate increases from 5 Mv/Seconds to 100 Mv/Seconds then the peak current is decreases from 6.07E-5 Ampere/Cm² to 1.61E-3 Ampere/Cm² and the charge stored in the film is changed from 1.76E-1 Coulomb to 1.20E-3 coulomb.



Fig 5: Current Vs Voltage Plots of 20 mM OPD coated PANI at 5 Mv/Seconds to 100 Mv/Seconds. For 20 mM OPD coated PANI as the Scan Rate increases from 5 Mv/Seconds to 100 Mv/Seconds then the peak current is decreases from 8.36E-5 Ampere/Cm² to 1.32E-3 Ampere/Cm² and the charge stored in the film is changed from 3.356E-1 Coulomb to 8.80E-3 coulomb.

Sr. No.	Scan Rate (v) in Volts/Sec	Sqrt Scan Rate $(v^{1/2})$	Peak Potential (Ep) Volts	Peak Current (Ip) Ampere/Cm ²	Log Ip	Charge(Q) in film in Coulomb
1	5	2.2361	0.187	0.00008363	-4.0776	0.3356
2	10	3.1623	0.133	0.00022820	-3.6417	0.1020
3	25	5.0000	0.143	0.00038480	-3.4148	0.0347
4	50	7.0711	0.154	0.00073520	-3.1336	0.0169
5	100	10.0000	0.171	0.00132400	-2.8781	0.0088

Table 5: Scan Rate Study of 20 mM OPD coated PANI:





Sr.No.	Slope	Intercept	Regression Constant	Relation between Ip and SR
1	0.00001	0.00007	0.9953	Straight line
2	0.000009	0.00002	0.9996	And Direct
3	0.00001	0.00003	0.9982	Relationship
4	0.00001	0.00002	0.9990	
5	0.00001	0.00004	0.9957	

Table: 6- Observation Table for Fig-6

From the above plots it is clear that during the pOPD formation as the Scan Rate increases faster the the peak currents are also increases linearly due to more potential gradient developed at the electrode surface against the the monomer concentration gradient forms at the electrode surface diffusing species and hence it follows the Randles-Sevcik equation and hence the more the scan rate more is the peak current.



Fig:7-Plot of Scan Rate Vs Peak Current -1.25 mM OPD Coated PANI to 20 mM OPD Coated PANI

Sr.no.	slope	exponent	Regression constant	Relation between Ip and sqrt SR
1	0.00002	1.9203	0.9968	
2	0.00001	1.9426	0.9989	Exponential
3	0.00001	1.8248	0.9998	
4	0.00001	1.8358	0.9994	
5	0.00002	1.7548	0.9815	

 Table:7- Observation Table for Fig-7



Figure 8:Plot of Scan Rate Vs Charge stored in film for -1.25 mM OPD Coated PANI to 20 mM OPD Coated PANI.

Sr.No.	Slope	Exponent	Regression Constant	Relation charge and SR
1	0.1202	-0.990	0.9968	Exponential
2	0.1646	-1.080	0.9989	
3	1.0528	-1.578	0.9998	
4	0.00001	-1.022	0.9994	
5	0.00002	-1.196	0.9815	

Table:8- Observation Table for Fig-8

#PPD coated PANI Study: #1.25 mm PPD coated PANI:

For 1.25 mM PPD Coated PANI, The peak current is increases from 6.39E-5 Ampere/cm² to 5.81E-4 Ampere/cm² and the charge in the film is decreases from 1.06E-2 coulomb to 6.85E-4 coulomb as the scan rate increases from 5 Mv/Seconds to 100 Mv/Seconds.



Fig 9: Current Vs Voltage Plots of 1.25 mM PPD coated PANI at 5 Mv/Seconds to 100 Mv/Seconds.

Sr. No.	Scan Rate	Sqrt Scan Rate	Peak Potential	Peak Current (Ip)	Log Ip	Charge(Q) in film in
	v	$\mathbf{V}^{1/2}$	(Ep) Volts	Ampere/cm ²		Coulomb
1	5	2.2361	0.117	0.00006396	-4.1941	0.010670
2	10	3.1623	0.124	0.00012720	-3.8955	0.005732
3	25	5.0000	0.140	0.00027510	-3.5605	0.002289
4	50	7.0711	0.154	0.00052820	-3.2772	0.001192
5	100	10.0000	0.159	0.00058100	-3.2355	0.000685

Table 9: Scan Rate Study of 1.25 mM PPD coated PANI:



Fig 10: Current Vs Voltage Plots of 2.50 mM OPD coated PANI at 5 Mv/Seconds to 100 Mv/Seconds.

Sr. No.	Scan Rate v	Sqrt Scan Rate $v^{1/2}$	Peak Potential (Ep) Volts	Peak Current (Ip) Ampere/cm ²	Log Ip	Charge(Q) in film in Coulomb
1	5	2.2361	0.145	0.00006135	-4.2122	0.022590
2	10	3.1623	0.139	0.00012520	-3.9024	0.012000
3	25	5.0000	0.145	0.00030090	-3.5216	0.004820
4	50	7.0711	0.160	0.00055320	-3.2571	0.002452
5	100	10.0000	0.162	0.00099410	-3.0026	0.001309

Table 10: Scan Rate Study of 2.50 mM PPD coated PANI

For 2.50 mM PPD coated PANI the peak current is increases from 6.12E-5 Ampere/cm² to 9.94E-4 Ampere/cm² and the charge in the film is decreases from 2.25E-2 coulomb to 1.30E-3 coulomb the scan rate increases from 5 Mv/Seconds to 100 Mv/Seconds. **#5 mM PPD coated PANI.**



Fig 11: Current Vs Voltage Plots of 5 mM OPD coated PANI at 5 Mv/Seconds to 100 Mv/Seconds.

Sr. No.	Scan Rate v	Sqrt Scan Rate v1/2	Peak Potential(Ep) Volts	Peak Current (Ip) Ampere/cm 2	Log Ip	Charge(Q) in film In Coulomb
1	5	2.2361	0.118	0.00006426	-4.1921	0.054340
2	10	3.1623	0.128	0.00012390	-3.9069	0.027170
3	25	5.0000	0.136	0.00027850	-3.5552	0.010860
4	50	7.0711	0.154	0.00053980	-3.2678	0.005452
5	100	10.0000	0.155	0.00098760	-3.0054	0.000281

Table 11: Scan Rate Study of 5 mM PPD coated PANI:

For 5 mM PPD coated PANI the peak current is increases from 6.42E-5 Ampere/cm² to 9.87E-4 Ampere/cm² and the charge in the film is decreases from 5.43E-2 coulomb to 2.81E-4 coulomb the scan rate increases from 5 Mv/Seconds to 100 Mv/Seconds.





Fig 12: Current Vs Voltage Plots of 10 mM PPD coated PANI at 5 Mv/Seconds to 100 Mv/Seconds.

For 10 mM PPD coated PANI the peak current is increases from 5.94E-5 Ampere/cm² to 9.61E-4 Ampere/cm² and the charge in the film is decreases from 1.55E-1 coulomb to 7.27E-3 coulomb the scan rate increases from 5 Mv/Seconds to 100 Mv/Seconds.

Sr. No.	Scan Rate	Scan Rate Squrt	Peak Potential	Peak Current (Ip) Ampere/cm ²	Log Ip	Charge(Q) in film in
	v	$v^{1/2}$	(Ep) Volts			Coulomb
1	5	2.2361	0.118	0.00005946	-4.2258	0.155800
2	10	3.1623	0.129	0.00012050	-3.9190	0.075010
3	25	5.0000	0.140	0.00026750	-3.5727	0.029370
4	50	7.0711	0.147	0.00049620	-3.3043	0.014530
5	100	10.0000	0.154	0.00096140	-3.0171	0.007277

Table 12: Scan Rate Study of 10 mM PPD coated PANI:

#20 mM PPD coated PANI:



Fig 13: Current Vs Voltage Plots of 20 mM PPD coated PANI at 5 Mv/Seconds to 100 Mv/Seconds.

Sr.	Scan	Sqrt Scan	Peak	Peak Current	Log Ip	Charge(Q)
No.	Rate	Rate $(v^{1/2})$	Potential	(Ip)		in film
	v		(Ep) Volts	In		In Coulombs
				Ampere/cm ²		
1	5	2.2361	0.112	0.00006515	-4.1861	0.336800
2	10	3.1623	0.124	0.00010240	-3.9897	0.158300
3	25	5.0000	0.139	0.00025840	-3.5877	0.062020
4	50	7.0711	0.152	0.00050070	-3.3004	0.030710
5	100	10.0000	0.153	0.00100300	-2.9987	0.015310

 Table 13: Scan Rate Study of 20 mM PPD coated PANI:

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Sr No.	Slope	intercept	Regression constant	Relationship
1	0.00001	0.00002	0.9994	linear
2	0.00001	0.00004	0.9961	
3	0.00001	0.00003	0.9982	
4	0.00009	0.00002	0.9960	
5	0.00001	0.000009	0.9998	

Table:14-Observation Table for Fig-14



Fig 15: Squrt Scan Rate Vs Peak Current for 1.25 mM PPD coated PANI to 20 mM PPD coated PANI.

Sr. No.	Slope	Exponent	Regression Constant	Relationship
1	0.00002	1.5436	0.9994	Exponential
2	0.00001	1.8583	0.9961	
3	0.00001	1.8248	0.9982	
4	0.00001	1.8358	0.9960	
5	0.00001	1.8572	0.9998	

Table 15: Observation Table for Fig-15

#Concentration and Charge Effect at specific scan rate for PPD (1.25 mM to 20 mM):

Concentration of PPD used	→ Scan Rate In Mv/Seconds	Charge (Q) at 5	Charge (Q) at 10	Charge (Q) at 25	Charge (Q) at 50	Charge (Q) at 100
1.25 mM		0.010670	0.005732	0.002289	0.001192	0.000685
2.50 mM		0.022590	0.012000	0.004820	0.002452	0.001309
5.0 mM		0.054340	0.027170	0.010860	0.005452	0.000281
10.0 mM		0.155800	0.075010	0.029370	0.014530	0.007277
20.0 mM		0.336800	0.158300	0.062020	0.030710	0.015310

 Table 16: Observation Table-Concentration and Charge in the film .



Fig:16 Plot of Concentration of PPD Vs Charge in the film at specific Scan Rate.

Sr.	Slope	Exponent	Regression Constant	Relationship
No.				
1	0.0174	-0.0158	0.9899	linear
2	0.083	-0.0088	0.9969	
3	0.0033	-0.0033	0.9972	
4	0.0016	-0.0016	0.9973	
5	0.0008	-0.0014	0.9535	

Table 17: Concentration and Charge in the film Observation Table.

#UV-Visible spectroscopic analysis:

The monomer solutions which are prepared in 0.5 M H₂SO₄ are subjected to UV-Visible spectroscopy and the polymer solutions are collected for the 20 mM concentration solution and at 5 Mv/Seconds scan rate after polymerization. The absorbance (OD) is found out by using 0.5 M H₂SO₄ as a blank solution.

#Aniline Monomer: Two UV-Visible spectroscopic peaks are found during UV-Visible scan of Monomer aniline. Peak 1 at 210nM with absorbance 2.5365 while the peak 2 is at 253 nM wavelength with absorbance 1.8789.

2) Indicates the presence of benzenoid ring with multiple bonds attached to it and p-p* transition.



Figure 17: UV-Visible spectrum of Aniline monomer in 0.5 M H₂SO₄.





The peaks are found at 200 indicates presence of benzenoid ring and 223 nM, Indicates the presence of benzenoid ring with multiple bonds attached to it and p-p* transition. With this one additional peak at 273 nM indicates the presence of Aniline Moity.

#PPD monomer: When PPD monomer is scanned under UV-visible spectrometer the two peaks are found peak 1 is major with high absorbance at 205 nM with 2.2510 absorbance while the peak 2 is minor with less absorbance 0.0940 at 260 nM wavelength. Presence of benzanoid ring with multiple bonds attached to it and p-p* transition.



#PPD polymer: PPD polymer solution when scanned under UV-Visible spectroscopy the peak 1 at 202 nM with absorbance 2.6816 and peak 2 at wavelength 234 with absorbance 0.7816 peak 3 at wavelength 280 with absorbance 0.4310 and peak 4 at wavelength 485nM with absorbance 0.2185. The peak at 485 indicates the polymerization of PPD that is PPD moity.



Figure 20: UV-Visible spectrum of PPD polymer

#OPD monomer: In OPD monomer the peaks at 210-270 are the peaks of related to benzanoid ring presence and loan pair presence.



Figure 21: UV-visible spectrum of OPD monomer.

#OPD polymer:The peaks in between 247 and 287 are related to benzanoid ring and additional peak at 492 nM is related to the pOPD polymer presence.





Conclusion:

It is found that the OPD and PPD monomers can easily be polymerized on PANI film at the same voltage range -0.2V to 0.75 V which used for PANI polymerization.Both for pOPD and pPPD polymers the peak current of cyclic voltammetric curve is increases with increase in the concentration of monomer and the charge stored in the polymer film is decreases with the increase in the concentration of the monomer.

1. The Peak currents for for OPD coated PANI is increases as the scan rate increases from 5 Mv/seconds to 100 Mv/seconds(8.36E-5 Ampere/cm² to 1.32E-3 Ampere/cm² for 20mM OPD coated PANI) and when the concentration of monomer increases from 1.25 mM to 20 mM the peak current increases further (1.19E-3 Ampere/cm² to 1.32E-3 Ampere/cm² for 100 RPM Scan Rate).

2. The Peak currents for for PPD coated PANI the peak current is increases as the scan rate increases from 5 Mv/seconds to 100 Mv/seconds(6.51E-5 Ampere/cm² to 1.00E-3 Ampere/cm² for 20mM PPD coated PANI) and when the concentration of monomer increases from 1.25 mM to 20 mM the peak current increases further (5.81E-4 Ampere/cm² to 1.00E-3 Ampere/cm² for 100 RPM Scan Rate).

3. The charge (Q) stored in the film for OPD coated PANI the charge is decreases as the scan rate increases from 5 Mv/seconds to 100 Mv/seconds(3.36E-1 coulomb to 1.53E-2 coulomb for 20mM OPD coated PANI) and when the concentration of monomer increases from 1.25 mM to 20 mM the charge decreases further (1.06E-2 coulomb to 3.36E-1 coulomb for 5 Mv/Seconds Scan Rate).

4. The charge (Q) stored in the film for PPD coated PANI the charge is decreases as the scan rate increases from 5 Mv/seconds to 100 Mv/seconds(3.3E-1 coulomb to 8.8E-3 coulomb for 20mM PPD coated PANI) and when the concentration of monomer increases from 1.25 mM to 20 mM the charge decreases further (2.54E-2 coulomb to 3.3E-1 coulomb for 5 Mv/Seconds Scan Rate).

5. The Uv-Visible analysis gives the characteristic peaks at 203 and 210 nM wavelength regarding the presence of benzanoid ring and p-p* transition while the peaks at 430-490 nM are related to the presence of polymer moity in the solution.

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