

# J E P T

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# Electrochemical Deposition and Characterization of Conjugated Copolymers of Thiophene and Aniline

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## Introduction

In recent years fully conjugated polymers with favorable physical properties has been much interested. Such materials must have atmospheric and thermal stability and desirable electrical properties. Electrically conducting conjugated polymers such as polythiophene, polyaniline and polypyrrole have attracted considerable attention [1-3]. But all of these conducting polymers are insoluble in common organic solvents which makes practical applications difficult.

Polythiophene are one of the most important class of conjugated polymers with wide range of applications, such as conducting films, electrochromics and field effect transistors [4-7]. Polyaniline has been studied extensively in the last few years due to its good environmental stability and facile synthesis. Such properties make this polymer suitable for a variety of applications [8-10].

Heterocyclic systems such as polythiophene has nondegenerate bond phases can be synthesized electrochemically due to small band gaps (2.1 eV), comparatively more stable and processible than

other non heteroatomic systems. Polyaniline, is another well-characterized heteroatomic polymer has nondegenerate bond phases that is quinoidal (Q) and aromatic (A) Figure 1 [11-12]. Quinoidal bond phases have higher energy conformation.

We choose copolymerization of thiophene and aniline due to both polymeric system have quinoi-

dal and aromatic phases Figure 2. Aniline is an electron donor, while thiophene is an electron acceptor having both properties within the same molecule. Thiophene rings with aromatic coupled structures are attractive due to their extending p-conjugated length, and closely packed rigid conformation [13]. We have observed that the presence of various substituent groups on the monomer unit 2 increases the solubility [14].

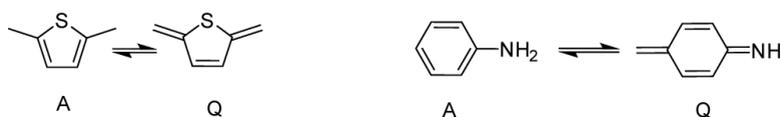


Fig. 1: Aromatic (A) and Quinoidal (Q) form of Thiophene and Aniline

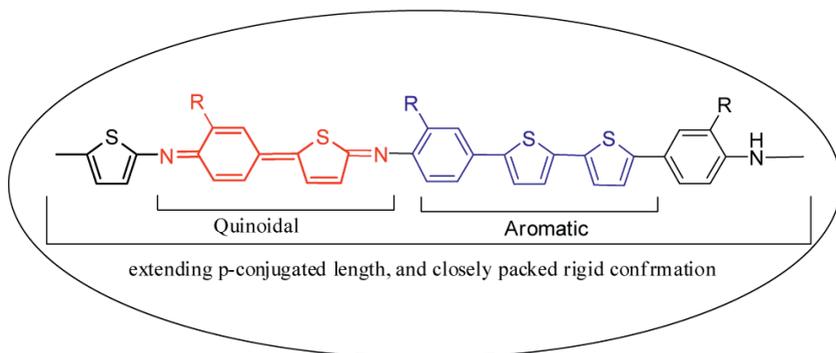
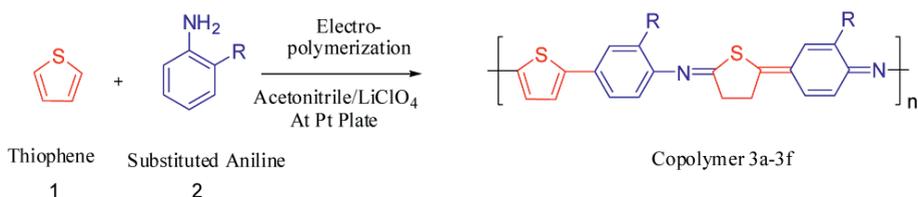


Fig. 2: Proposed structure of copolymer formed from Thiophene Aniline monomer on platinum plate coupling by electropolymerization.



Scheme 1: Schematic reactions for route synthesis of copolymer of Thiophene with Aniline derivative

The aim of this study, is to obtain a co-polymer of two organic compound having totally different properties by applying different electrochemical conditions such as potential, solvent. There are few studies about the synthesis of copolymers of thiophene and aniline or their derivatives prepared by electrochemical and chemical [15-16] methods. Electropolymerization has some advantages. It is a simple and relatively inexpensive method, it permits synthesis of copolymer without using any oxidizing agent. Application of electricity as a non-conventional energy source for activation of reactants in suitable solvents has now gained popularity over the usual homogeneous and heterogeneous reactions. In addition by adjusting conditions (potential, time, temperature), both powders and films can be obtained [17].

(Monomer 2) =  $C_6H_4NH_2$ ,  $o-ClC_6H_4NH_2$ ,  
 $o-CH_3C_6H_4NH_2$ ,  $o-OCH_3C_6H_4NH_2$ ,  $o-OHC_6H_4NH_2$ ,  
 $NH_2C_6H_4NH_2$ ,  $N-C_2H_5C_6H_5NH$ ,  $N-CH_3C_6H_5NH$

## Experimental Section

### Chemicals

The monomer used in preparation of polymer are shown in Table 1 Aniline GRgrade (Merck), thiophene (Aldrich). per-chloric acid from (Merck) was used for providing acid medium and  $LiHO_4$  (Aldrich) was used as a supporting electrolyte.. Acetonitrile and ethylacetate were obtained from LobaChemie. P-chloroaniline and o-chloroaniline from CDH and other aniline derivative obtained from Lobachemie. These chemical were used without further purification. Water used was double distilled.

### Electrochemical Synthesis of copolymer

Electrochemical experiments were carried out with potentiostatgalvonostat system. A

three-electrode system was used. The working and the counter electrodes were platinum plates having a surface area of  $1.2\text{ cm}^2$ . Electrochemical measurements were carried out at room temperature in a three-electrode beaker type undivided cell with the reference electrode (Hg, HgCl saturated calomel) in acetonitrile. Controlled potential electrolysis was carried performed to a solution of Thiophene with aniline in 4:1 ratio containing  $LiClO_4$  (7 mmole) as supporting electrolyte in an undivided cell equipped with a magnetic stirrer. The polymerization current was supplied by dc power supply. The progress of electrolysis was followed by periodically decrease in current with time. The electro-copolymerization of thiophene with aniline afforded purple black precipitates as products. Obtained product was extracted with ethyl acetate and supporting electrolyte was filtered off and obtained residue was purified using distill water. All reactions were performed under same electrolytic condition up to 8-10 h and eight samples were prepared such as poly Thio-co-Ani.

### Characterization technique

All experiments were carried out in a conventional electrochemical cell which consists of platinum plates in the form of flattened sheet as working as well as counter electrode and saturated calomel electrode (SCE) as reference electrode. Cyclic voltammetry was performed using a Versa stat 3200(USA) CH instrument with a three electrode system. Infrared spectra were recorded on a Nicolet 460 IR spectrometer with KBr pellet. The scanned wavenumbers range from  $4000$  to  $400\text{ cm}^{-1}$ . X-ray diffraction patterns were obtained for thio coanipowders, using a XPERT-PRO diffraction system start position [ $^{\circ}2\theta$ ] 5.0084 to end position [ $^{\circ}2\theta$ ] 69.9824 using a graphite monochromator with K-Alpha 1 ( $\lambda = 1.54060\text{ \AA}$ ) radiation, reflection mode, step of  $0.0170^{\circ}$ , step time 25.19. The microstructure and surface morphol-

ogy of synthesized polymer was studied by scanning electron microscope (SEM) using electron probe microanalyser. The solubility tests were carried out at room temperature by using 1 mg sample and 1 mL solvent.

## Results and discussion

Electrochemical synthesis of poly Thio-co-Ani has been carried out in electrochemical cell in which Thiophene is used as monomer I and Aniline (Aniline derivative) as monomer II and formed copolymer product is listed as (3a-3f) (Scheme 1). Controlled Potential Electrolysis (CPE) is performed in electrochemical synthesis of copolymer. Current-Potential data of Electro-copolymerization of thiophene with aniline and its derivative are given in (Table 1, Entry 1-10). In Mechanistic pathway for the formation of poly Thio-co-Ani monomer units such as Thiophene and Aniline electrochemically oxidized to its radical cation by giving a electron. These radical cation combined with each other and formed oligomer of thiophene-aniline by removal of two moles of proton. This oligomer undergo oxidation and chain elongation process, long chain of copolymer of thiophene with aniline is formed (Scheme 2). Poly Thio-co-Ani are

soluble in different organic solvents such as NMP, DMSO, Chloroform and Benzene. Copolymer is insoluble in polar solvent such as Ethanol and Acetone (Table 2).

## Electrochemical properties

All the electrochemical measurements were carried out with Electrochemical Analyzer CH Instruments at room temperature in a three electrode system. Electropolymerization of thiophene and

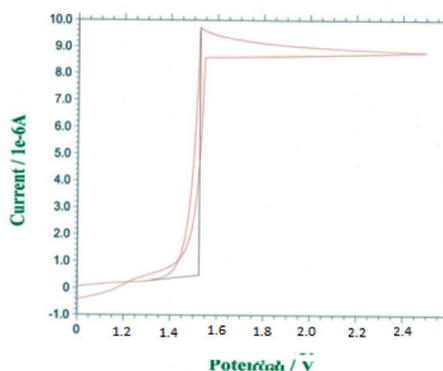


Fig. 3: The cyclic voltammogram of poly Thio-co-Ani in an acetonitrile solution containing  $\text{LiClO}_4$ . The scan rate was 100 mV/s.

Entry	Monomer I	Monomer II	Potential/V	Current	Time/M	Yield%
1	$\text{C}_4\text{H}_4\text{S}$	$\text{C}_6\text{H}_5\text{NH}_2$	1.7	18	240	52
2	$\text{C}_4\text{H}_4\text{S}$	<i>o</i> - $\text{ClC}_6\text{H}_4\text{NH}_2$	1.75	17	360	40
3	$\text{C}_4\text{H}_4\text{S}$	<i>o</i> - $\text{CH}_3\text{C}_6\text{H}_4\text{NH}_2$	1.65	22	300	48
4	$\text{C}_4\text{H}_4\text{S}$	<i>o</i> - $\text{OCH}_3\text{C}_6\text{H}_4\text{NH}_2$	1.60	31	220	50
5	$\text{C}_4\text{H}_4\text{S}$	<i>o</i> - $\text{OHC}_6\text{H}_4\text{NH}_2$	1.70	29	260	48
6	$\text{C}_4\text{H}_4\text{S}$	<i>o</i> - $\text{NH}_2\text{C}_6\text{H}_4\text{NH}_2$	1.62	30	320	50
7	$\text{C}_4\text{H}_4\text{S}$	<i>p</i> - $\text{ClC}_6\text{H}_4\text{NH}_2$	1.65	20	400	56
8	$\text{C}_4\text{H}_4\text{S}$	<i>p</i> - $\text{CH}_3\text{C}_6\text{H}_4\text{NH}_2$	1.65	18	360	58
9	$\text{C}_4\text{H}_4\text{S}$	<i>p</i> - $\text{OCH}_3\text{C}_6\text{H}_4\text{NH}_2$	1.50	31	420	58
10	$\text{C}_4\text{H}_4\text{S}$	<i>p</i> - $\text{OHC}_6\text{H}_4\text{NH}_2$	1.54	34	340	62

Tab. 1: Current-Potential data of Electro-copolymerization of thiophene with aniline and its derivative via radical and radical cation coupling mechanism

aniline has been found to be difficult because of large difference in their oxidation potentials. Aniline polymerizes electrochemically in a dilute aqueous acidic solution at a potential of 0.800 eV. On the other hand, thiophene polymerizes in a nonaqueous or highly concentrated acidic solutions at a high anodic potential of 2.00 eV. This problem can be solved by adjusting polymerization potentials according to concentration of monomers. Due to the large difference in the electrooxidation potentials of aniline and thiophene results formation of polyaniline film with polythiophene film [18]. To avoid this, aniline–thiophene copolymers were deposited experimentally at constant potential 1.7 eV versus callomel as a reference electrode.

### Cyclic Voltammogram

Cyclic voltammetry (CV) was employed to assay the electro activity of the compounds and determine the oxidation–reduction peak potentials Figure 3. CV measurements were carried out at a potential scan rate of 0.1 V/s. The system consisted of a CV cell containing Pt plate as the working electrode, as well as counter electrode, and callomel as the reference electrode in the employed electrolyte solution. Cyclic voltammetry curve was drawn corresponding in the electrode potential range of 1.00–2.40 eV. One oxidative peak was observed at potential range 1.60 eV and absence of cathode peak implies that electrochemical oxidation of thiophene with aniline is

solvent	NMP	DMSO	CHCl <sub>3</sub>	Benzene	THF	TFA	Ethanol	Acetone
solubility	++	+++	+	+	+	+	-	-

Tab. 2: Solubility of poly-co-thio-o-Ani in different organic solvent

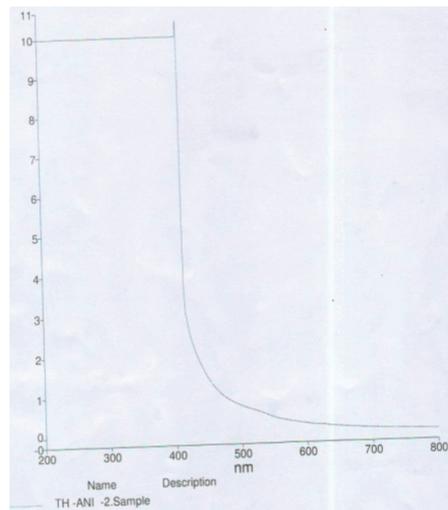
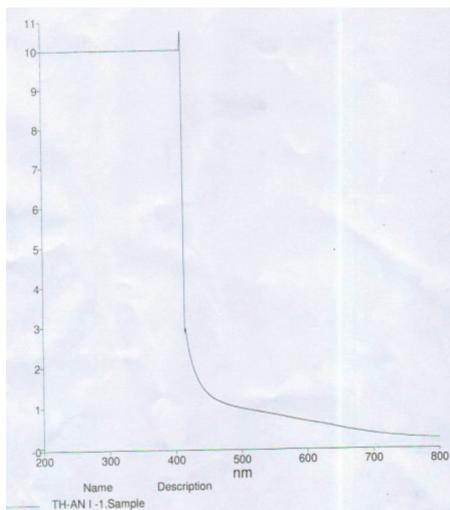
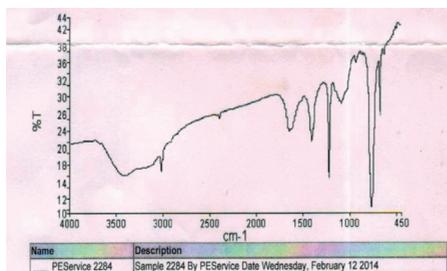


Fig. 4: UV–Visible spectroscopic characterization of electrocoated copolymer powder of poly-thio-co-aniline (4a) and poly-thio-co-o-toluidine (4b)

an irreversible process. When electrode potential reached at 1.6 V current was maximum. A cyclic voltammogram is slightly different behavior of its monomers during electrooxidation. Oxidation peak indicates the formation of aniline radical cation. Polyaniline coating formation takes place at lower potential than thiophene which disturbs the polythiophene coating formation. This provide information about the possibility for thiophene-aniline copolymerization.

### UV-Vis Spectra

Figure 4 depicts the UV-Vis absorption spectra of copolymer, absorption maxima corresponding to benzenoid, and quinoidal electronic transitions ( $\pi-\pi^*$ ) are observed at about 450 nm - 700 nm.

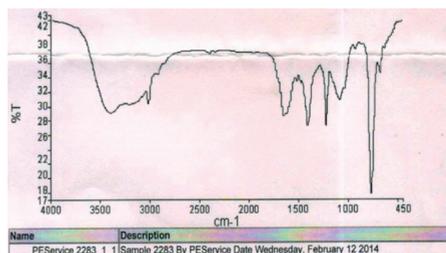


PeakName	X	Y
11	627.83	37.56
10	669.22	26.65
9	758.86	10.33
8	928.44	36.13
7	1087.39	28.95
6	1215.76	15.74
5	1403.97	22.17
4	1650.84	23.92
3	2400.49	26.2
2	3020.07	16.61
1	3410.64	16.12

Imino nitrogen ( $n-\pi^*$ ) transitions were registered between 650 nm and 800 nm. Spectra shows absorption bands at higher wavelength (red-shifting) which is observed due to the presence of different oxidation states into the polymeric chain, like the quinoidal units, the semi-planar and non-branched structure that leads to a more conjugated and energetically more stable polymer Figure 2. Copolymer of thiophene and aniline leads to red shift of absorption maximum at the room temperature suggesting a greater p-conjugation and more ordered structure [19].

### FT-IR of poly-thio-co-ani

Partial information on the structure of the copolymer was obtained from IR spectroscopy (Figure 5a/b). As there are several similar bands in both spectra which, in accord with similar series as poly-co Thiophene-Aniline and poly-co Thiophene-o-Anisidine. A very broad peak at 3400-3410  $\text{cm}^{-1}$



PeakName	X	Y
8	670.36	34.58
7	763.83	17.71
6	1080.55	31.02
5	1216.08	27.27
4	1402.92	27.32
3	1647.49	28.75
2	3020.32	30.45
1	3400.03	29.13

Fig. 5: FT-IR spectra of co-polymers of poly-Thio-co-Ani (5a) and poly-thio-co-o-toluidine (5b)

is characteristic of the N-H stretching vibration and suggests the presence of secondary amino groups [20]. The lower frequency mode at  $1495\text{ cm}^{-1}$  indicates the presence of benzenoid ring units. Two bands of middle intensity are also observed at  $1216$  and  $1215\text{ cm}^{-1}$ , which can be ascribed to the C-N stretching vibration in the quinoid imine units and the C-N stretching vibration in the benzenoid imine units [21]. The bands in the region of  $758/763\text{ cm}^{-1}$  are due to the C-H out-of-plane deformation which are characteristic of the 1,4-disubstituted benzene as well as of 2,3,5-trisubstituted

thiophene [22]. The presence of para-disubstituted phenylene rings and  $\alpha$ - $\alpha$  coupled thiophene rings indicates that the coupling rings produce preferentially linear elements of chain.

### X-Ray Diffraction Technique

Figure 6a/b shows the XRD patterns of poly-thio-co-ani. It can be seen that conjugated polymers (thiophene-Aniline) with linear molecular geometry gives XRD peak in the low-angle region  $12^\circ$ - $22^\circ$ . The peak of about  $4.04^\circ$  is reasonable for a stack-

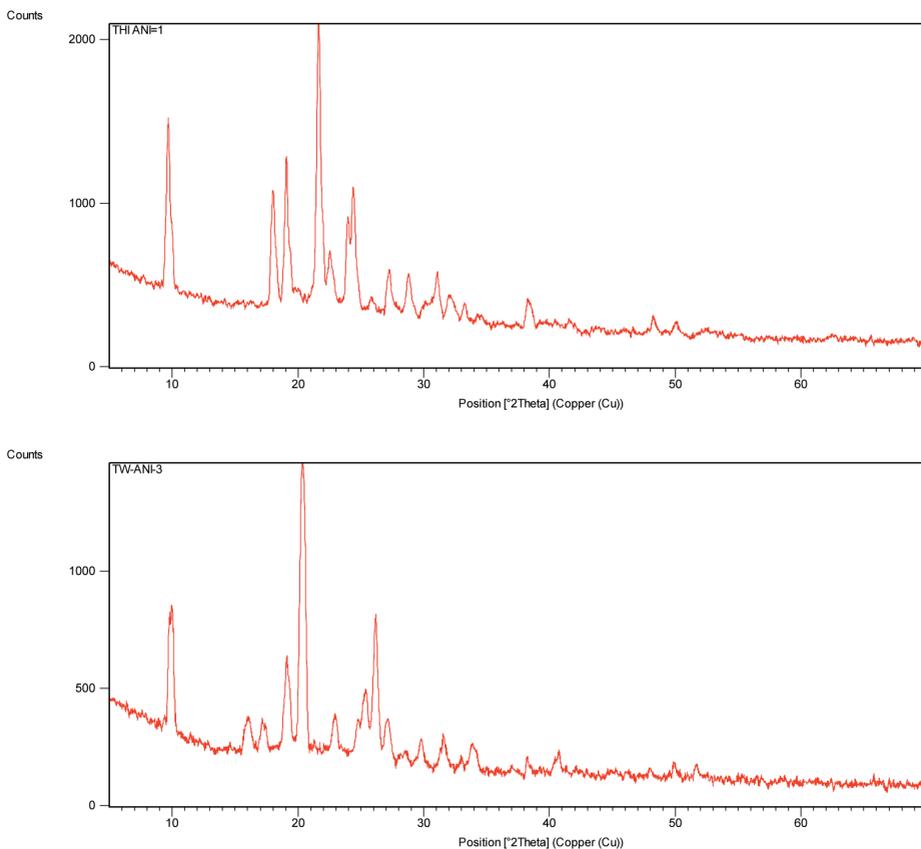


Fig. 6: XRD patterns of co-polymers of poly-Thio-co-Ani (6a) and poly-thio-co-o-toluidine (6b) in powder form

ing distance of thiophene-based conjugated polymers. Peaks at  $25^\circ$  and  $30^\circ$  corresponds to XRD signals of conducting glass, suggesting that there are an amorphous packing of the polymer molecules. These studies revealed that the copolymers have extended ranges of stability even at very positive applied potentials compared with homopolymers.

### Surface morphologies of the copolymer (poly-thio-co-ani)

The surface morphological properties of poly-thio-co-ani are obtained by scanning electron microscopy (SEM). SEM photographs of the powder forms are

shown in Figure 7. After the copolymers are electrochemically synthesized onto Platinum, the copolymeric materials are scraped from the Pt plate and the measurements are carried out. According to the SEM images, poly-thio-co-ani has a perforated surface with holes approximately 2–3  $\mu\text{m}$  in diameter (Figure 7a/b). This morphology is distinctly different from that of the polythiophene homopolymers.

Polymer surface is homogeneous. The perforated structure supplies a larger surface area in a small volume, which improves the performance. Similarly, this morphology poly-thio-co-ani is expected to increase the functionality.

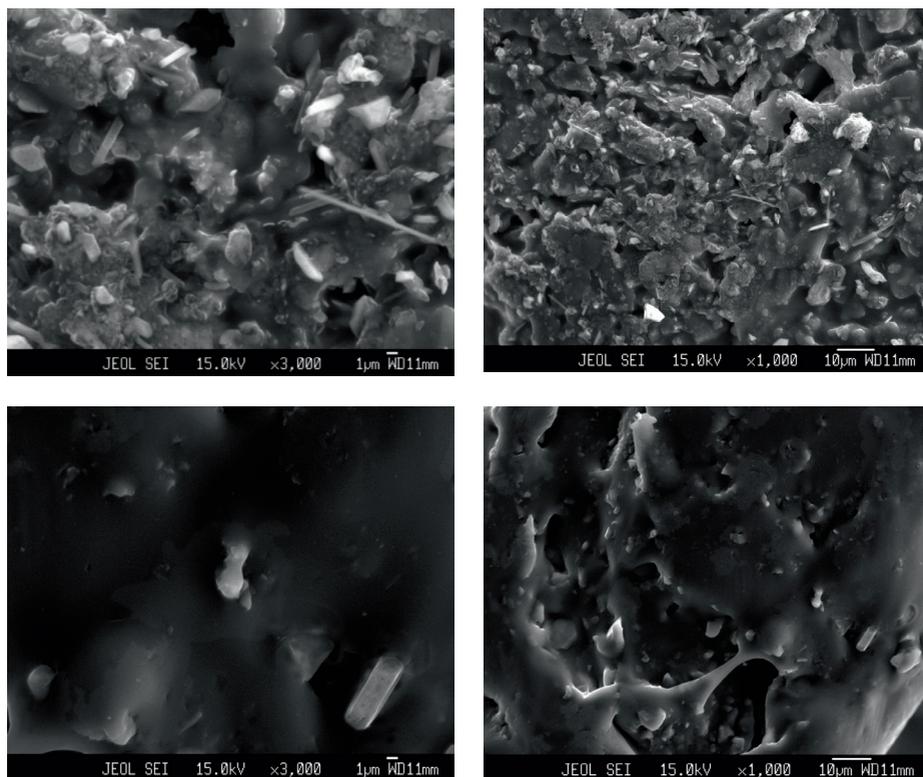
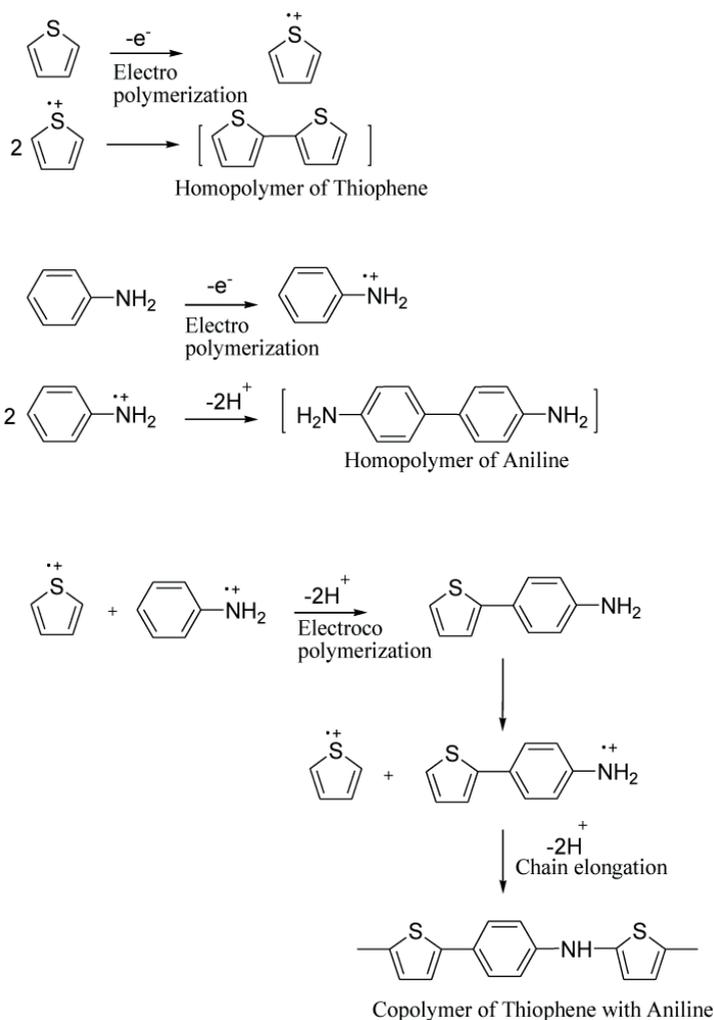


Fig 7: SEM images of the poly-co-thio- Ani (7a) and poly-thio-co-o-toluidine (7b) with different magnification and cross-section



Scheme 2 Plausible mechanistic pathway for the copolymer of Thiophene with aniline

## Conclusion

It can be concluded that copolymer of polythiophene and polyaniline having nondegenerate bond phases can be synthesized electrochemically. Furthermore, it was found that the inser-

tion of aliphatic chain to the polymer backbone significantly increases the solubility of the product that notably affects the thermal stability of the system. Synthesis of these copolymers takes place between 1.4 and 1.9 V due to the presence of aniline units within the polythiophene

chains. The range of stability of these copolymers is considerably extended. Synthesize copolymer have extended p-conjugated length and closely packed conformation used in different field. The copolymerization reaction is strongly affected by the monomer concentration. Electrochemical method is more convenient to synthesize the copolymer films.

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