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Synthesis, Characterization and Corrosion Behavior of Isomers of Conducting Poly–Toluidine on Mild Steel in Acid Medium

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Abstract: ***Isomers of Poly-toluidine have been synthesized using chemical oxidation method at 0°C using potassium dichromate as oxidant and hydrochloric acid as dopant. The synthesized polymers were characterized using IR, UV, TGA, DTA, XRD and SEM. The AC conductivity of POT, PMT, and PPT showed that they were semiconducting in nature. The corrosion behavior of the polymer on mild steel in acidic medium was studied by using electrochemical techniques .The studies showed that the inhibition increased with increase in concentration and reaches an optimum efficiency. The study further revealed that the inhibition efficiency followed the order PMT>PPT>POT. Polarization studies indicated that these three polymers act as mixed type corrosion inhibitors and EIS study showed that the dominant coating mechanism follows kinetic controlled corrosion mechanism.***

Keywords: poly toluidine, AC conductivity, mild steel, corrosion inhibition, inhibition efficiency,

1. Introduction

Corrosion is an electrochemical phenomenon and is accompanied by the flow of electrical phenomenon and is accompanied by the flow of electrical current. One can minimize the rate and quantum of corrosion only by providing a suitable environment in which current cannot flow at interfaces. The best corrosion protection is to build a barrier separating corrosion protection is to build a barrier separating the metal from its environment [1]. Protection of mild steel MS from corrosion is a matter of practical importance as it has favorable properties and high speed of fabrication [2-4]. The most efficient corrosion inhibitors are organic compounds containing electronegative functional groups and π electrons in their triple or conjugated double bonds [5],[6]. High electron density of the sulphur and nitrogen atoms present in hetero compounds helps the organic molecule to get chemisorbed onto the metal surface [7]. The earlier studies [1],[8-11] have shown that the inhibitive properties of conducting polymers like polyaniline and its derivatives are good corrosion inhibitors on iron in acid solution due to the presence of π electrons, quaternary nitrogen atom and large molecular size. Lu et al [12] and Camalet et al [13] deposited PANI films on MS from aqueous oxalic acid. Several ring substituted anilines namely *o*-toluidine, *m*-toluidine, *o*-anisidine and *o*-chloro aniline were electro deposited on passivated surfaces [14]. Substituted and unsubstituted PANI [15], [16], poly amino quinone [17] and poly [diphenylamine] [18] have been reported for their corrosion inhibition properties. Anti-corrosive properties of PANI, Poly *m*-toluidine and Poly (aniline-co-2-toluidine) coatings on stain steel were studied by Evrim Hur et al [19]. Poly *o*-toluidine coating as a corrosion protective agent for copper in chloride environment was examined by M.Mobin et al [20].The mechanisms postulated for conducting polymer to act as a corrosion inhibitor may include formation of a protective barrier layer, inhibition by the adsorption of organic species and the anodic passivation achieved when the corrosion potential is shifted to more positive values under the influence of charge transfer from the conducting polymer [21], [22].

To the best of our knowledge, there was no report in the literature dealing with the corrosion protection behavior of isomers of conducting poly toluidine synthesized by chemical oxidation method on MS in acid medium. So an attempt has been made to investigate the corrosion protection behavior of chemically synthesized conducting poly (*ortho*-, *meta*-, and *para* toluidine*)* over MS in acidic environment using potentiodynamic polarization measurement and electrochemical impedance spectroscopy (EIS) measurements.

2. Experimental Method

2.1. Synthesis and Characterization of the Polymers

0.1M monomers [*ortho*-, *meta*-, and *para*-toluidine] were subjected to chemical oxidation method with potassium dichromate as oxidant and 1M HCl as dopant for 12 hrs at 0°C [23],[24].The resulting precipitated polymers were filtrated and washed with excess of water, methanol, and acetone to remove excess of acid and the oxidant present in the polymers and they were dried at 500°C for 24hr.

The FT-IR spectrums of polymers were recorded by Thermo Nicolet, Avatar 370 spectrophotometer. The spectrum of the dry polymer powder in KBr pellet was recorded from 500 cm^{-1} to 4000 cm^{-1}. UV-VIS-NIR spectra of polymers dissolved in DMSO solvent were obtained using Varian, Cary-5000 spectrophotometer in the range of 200–2500 nm. Thermo gravimetric analysis [TGA] was carried out in nitrogen atmosphere at a heating rate 10^0C/min up to 750^0C temperature by PerkinElmer, Diamond TG/DTA analyzer. X-ray diffraction [XRD] scan was done with Bruker AXS D8 advance diffractometer at room temperature using Cu Kα [λ = 1.5406 ˚ A].

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2.2 Anticorrosion Studies

Potentiodynamic polarization measurements were carried out with three electrode system. Mild steel with exposed area of 1cm^2 was used as the working electrode. Platinum foil and the calomel electrodes were used as counter electrodes and reference electrode, respectively. These electrodes were immersed in 100ml of 1M HCl with and without inhibitors. The potentiodynamic polarization measurements were carried out at a sweep rate of 2mV/s using CH electrochemical analyser (Model CHI 608B) instrument. Impedance measurements were carried out in the frequency range of 10 kHz to 0.01Hz using CH electrochemical analyser [Model CHI608B] .The solution resistance and total resistance were obtained from the low-frequency and high frequency intercepts on Z' –axis of Nyquist plot respectively. The difference between R_t and R_s values give the charge transfer resistance R_{ct} value. The surface preparation of the mechanically abraded specimens was carried out using different grades of silicon carbide emery paper (up to 1200 grit) and subsequent cleaning with acetone and rinsing with double distilled water were done before each experiment. Before each potentiodynamic polarization (Tafel) and EIS (Nyquist) studies, the electrode was allowed to corrode freely and its open circuit potential (OCP) was recorded as a function of time up to 20 min, which was sufficient to attain a stable state. After this, a steady state of OCP corresponding to the corrosion potential (Ecorr) of the working electrode was obtained. The potentiodynamic measurements were then started from cathodic to the anodic direction, $E = E\text{corr} \pm 250\text{mV}$, at a scan rate of 10mVs^{-1}.The linear Tafel segments of the anodic and cathodic curves were extrapolated to obtain Ecorr and corrosion current density (Icorr).The inhibition efficiency was evaluated from the measured Icorr with and without inhibitor using the relationship

$$\text{IE}\ [\%] = \frac{I^0_{corr} - I_{corr}}{I_{corr}}\ [1]$$

Where I^0_{corr} is the corrosion current density without inhibitor and I_{corr} is the corrosion current density with inhibitor.

The potentiodynamic polarization and EIS data were analyzed and fitted using graphing and analyzing impedance software, Nova 1.4. Fresh solution and fresh steel samples were used after each sweep. The charge transfer resistance (Rct) was obtained from the diameter of the semicircle of the Nyquist plot. The inhibition efficiency of the inhibitor has been found out from the following equation:

$$\text{IE}\ [\%] = \frac{R_{ct} - R^0_{ct}}{R_{ct}} \text{ X } 100\ [2]$$

where R^0_{ct} and R_{ct} are the charge transfer resistance in the absence and in the presence of the inhibitor.

3. Results and Discussion

3.1Spectral and Thermal Studies

The representative FT-IR spectra of three Polymers POT, PMT, and PPT are given in Figure 1. The presence of strong bands at 1598cm$^{-1,}$ 1595cm-^{1}, 1604cm^{-1} and at 1487cm^{-1}, 1481cm^{-1} in POT, PMT and PPT respectively shows that they consist of amine (benzenoid) and imine (quininoid) units. A N-H stretching vibration is observed at 3398 cm^{-1}, 3349 cm^{-1} and at 3425 cm^{-1} respectively for POT, PMT and PPT.

The UV-VIS-NIR absorption spectrums of all the polymers dissolved in DMSO are shown in Figure 2.The first absorption peaks for POT,PMT ,PPT are observed at 312 nm,328 nm,276 nm which is assigned to π-π* transition and second absorption peaks at 582 nm,562 nm ,449 nm is assigned to n-π* transition and insulating pernigraniline phase of the polymer. The peaks at 806 nm for POT, 814 nm for PMT and 765 nm for PPT correspond to conducting emeraldine phase. The thermal stability of the three polymers is shown in Figure 3.The thermal behavior of the three polymers is similar and exhibits a three stage decomposition pattern. The first step is due to loss of water, and dopant, the second due to oligomers and the polymer is decomposed in the third step.POT is thermally more stable [630^{0}C] than that of PMT [480^{0}C] and PPT [430^{0}C].

3.2 Conductivity Studies

AC conductivity for POT, PMT and PPT are 1.89566 X 10^{-05} S/cm, 6.51588 X 10^{-07} S/cm and 1.29725 X 10^{-08} S/cm at room temperature and found to increase with increase in temperature.

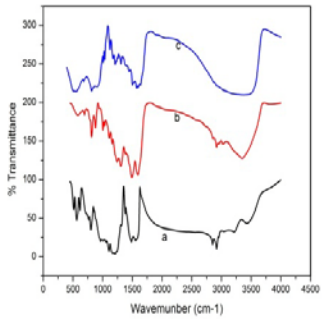

Figure 1: FT IR of [a] POT [b] PMT [c] PPT

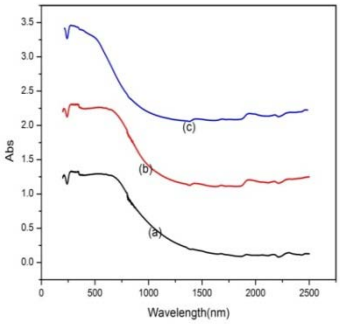

Figure 2: UV-VIS of [a] POT [b] PMT [c] PPT

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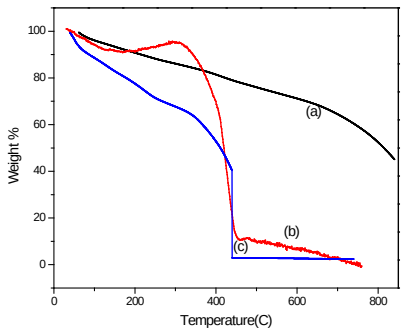

Figure 3: Thermograms of [a] POT [b] PMT [c] PPT

3.3. Potentiodynamic Polarization Studies

The potentiodynamic polarization curves of MS in 1MHCl with the addition of various concentrations of POT, PMT, and PPT are shown in Figures 4[a], [b], and[c], respectively. The corrosion kinetic parameters such as corrosion current density *I*corr, corrosion potential *E*corr, anodic Tafel slope *ba*, and cathodic Tafel slope *bc*, and inhibition efficiency IE, deduced from the curves are given in Table 1. The corrosion current density values decreases from 4.26µA/cm^{-2} for the blank acid to 1.31, 1.5, and 2.36 μAcm−2 for POT, PMT, and PPT, respectively, for the addition of 1000 ppm of three polymers resulting in 40%, 56%, and 40.2% of inhibition efficiency. It is clear that the *I*corr values decrease with the presence of three polymers which indicated that polymers are adsorbed on the metal surface and hence inhibition occurs. As the concentrations increases from 100ppm to 1000ppm for POT, PMT, PPT the IE increases from 59% ,48.9%, 39.8%, reaches a optimum efficiency of 63.5%, 52% and 47%, at 800 ppm,400 ppm and 400ppm respectively and then decreases. The *E*corr, *ba*, and *bc* values do not change appreciably with the addition of the inhibitors indicating that the inhibitors are not interfering with the anodic dissolution or cathodic hydrogen evolution reactions independently but acts as a mixed type of inhibitor [10],[23],[24]. The present results confirm that the isomers of poly toluidine on MS act as a highly protective layer, which is mainly attributed to the presence of π electrons in aromatic ring coexisting with quaternary nitrogen atom and large molecular size [25]. Moreover, the higher corrosion protection offered by polytoluidines on MS may be due to the fact that the deposited polymer is strongly adhering and uniformly covers the entire electrode surface. Based on the corrosion protection mechanism put forth by several researchers [26] the polymer accepts electrons from the metal and gives them to oxygen. This reaction generates the formation of a passive layer at the polymer/metal interface which lowers the corrosion rate and shifts the corrosion potential to more positive values. The higher inhibitive property of Poly [*m*-toluidine] is also due to the presence of π electrons, quaternary nitrogen atom and the larger molecular size which ensures greater coverage of the metallic surface.

Table 1: Potentiodynamic polarization parameters for mild steel in 1M HCl with and without the inhibitors at different concentrations

Inhibitor Conc ppm	POT					PMT					PPT				
	ba (mV/ dec)	bc (mV/ dec)	-Ecorr (mV)	Icorr (µA/cm^2)	IE %	ba (mV/ dec)	bc (mV/ dec)	-Ecorr (mV)	Icorr (µA/cm^2)	IE %	ba (mV/ dec)	bc (mV/ dec)	-Ecorr (mV)	Icorr (µA/cm^2)	IE %
Blank			572	4.26				572	4.264				572	4.26	
100	7584	11515	537	3.42	40	8875	8645	520	4.05	59	8574	5999	527	2.52	48.9
200	7752	10557	533	2.81	44	8574	9645	517	1.99	62	8465	6461	525	2.45	50.6
400	7927	10117	520	2.69	47	8032	9638	510	1.98	64	8182	5994	521	2.21	52
600	7780	9987	510	2.36	43	8707	8144	508	1.89	65	8244	6170	531	1.8	43
1000	7470	9678	504	1.98	33	8909	7812	504	1.64	69	8102	5547	535	1.74	41
1000	8501	6739	507	1.5	40	8878	9043	499	1.31	56	8501	6739	537	1.5	40.2

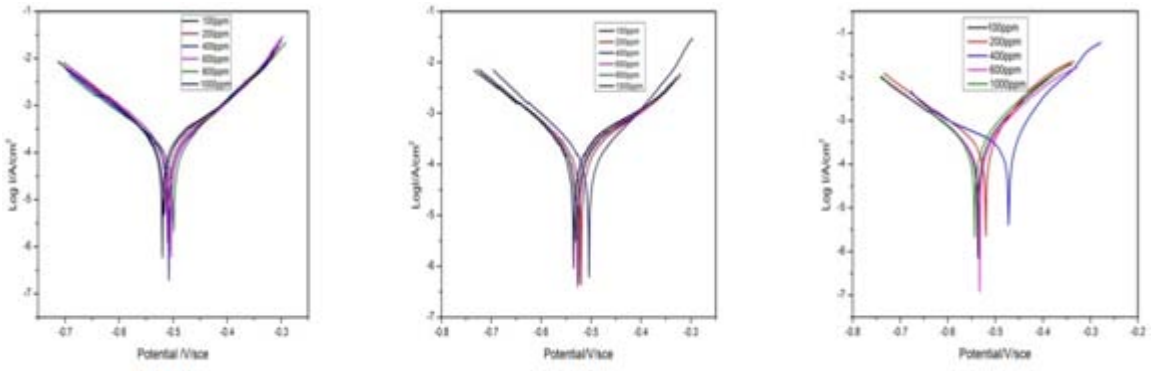

Figure 4: Potentiodynamic polarization curves obtained for mild steel in 1MHCl in the presence of different concentrations of (a) POT (b) PMT (c) of PPT

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3.4 Electrochemical Impedance Spectroscopy

The Nyquist impedance plots were analyzed by fitting the experimental data to a simple equivalent circuit model. In this equivalent circuit, Rs is the solution resistance, Rct is the charge transfer resistance, and CPE is a constant phase element, which is placed in parallel to charge transfer resistance element. Thus, in these situations pure double layer capacitors [Cdl] are better described by a transfer function with constant phase elements to give a more accurate fit. The Nyquist plots of MS in 1M HCl in the presence of various concentrations of POT, PMT, and PPT are depicted in Figures 5[a], 5[b], and 5[c], respectively. The Nyquist spectra of isomers of polytoluidines at various concentrations showed depressed semicircle, representing the corrosion process of the system, that is, the charge transfer resistance due to the metal corrosion and the double layer capacitance of the liquid/metal interface. The depressed semicircles in the Nyquist plots were probably due to the surface heterogeneity or corrosion products of the metal substrate. The element at high frequency region is related with the charge transfer resistance of system; elements at low frequency region may represent adsorption of ions, for example, Cl− which is the initial step of anionic or solvent exchange between polymers and solution during equilibration in 1M HCl [27],[29]. The Nyquist spectra of isomers of poly toluidine revealed one-time constant behaviour, which is attributed to the charge transfer resistance of corrosion process [30]. The higher impedance is probably due to an area effect where the inhibitor is blocking access of the aggressive electrolyte to the reactive metal surface [31].The impedance values are given in Table 3. The charge transfer resistance is increased from 30.443Ωcm^2 for blank solution to 133.11, 263.96, and 144.64Ωcm^2 upon addition of 800ppm, 400ppm, 400ppm of POT, PMT, and PPT resulting in 76.89, 88.46, and 78.8% inhibition efficiency, respectively and decreases there on. The increase in R_{ct} value is attributed to the formation of an insulating protective film at the metal/solution interface [32, 33]. The double layer capacitance decreases from 9.669 μFcm^{-2} to 2.236, 6.0071, and 5.012 μFcm^{-2} on the addition of 800ppm, 400ppm, and 400ppm of POT, PMT, and PPT respectively. The initial decrease in Cdl value from blank solution to inhibitor containing electrolyte is due to a decrease in the local dielectric constant, while further decrease in Cdl with increasing concentrations of the inhibitor is due to increase in the thickness of the electrical double layer. The enhanced corrosion protection of mild steel by isomers of polytoluidine is explained on the basis of molecular adsorption. The polymers inhibit corrosion by controlling both the anodic and cathodic reactions. In acidic solution, the polymer molecules exist as protonated species [34] and these protonated species adsorb on the cathodic sites of MS and decrease the evolution of hydrogen. The adsorption on anodic sites occurs through long π-electrons of aromatic rings [benzenoid and quinoid] and lone pair of electrons of nitrogen atoms, which decreases the anodic dissolution of mild steel [35]. It is well known that the species having high molecular weight and bulky structure may cover more area on the active electrode surfaces [36]. Nyquist plots are more informative to investigate the dominant coating corrosion mechanism, eg, kinetic [semicircle] and diffusion controlled [straight line] mechanisms. The semicircle shape of these plots indicates that the dominant coating mechanism is kinetic controlled corrosion [37].

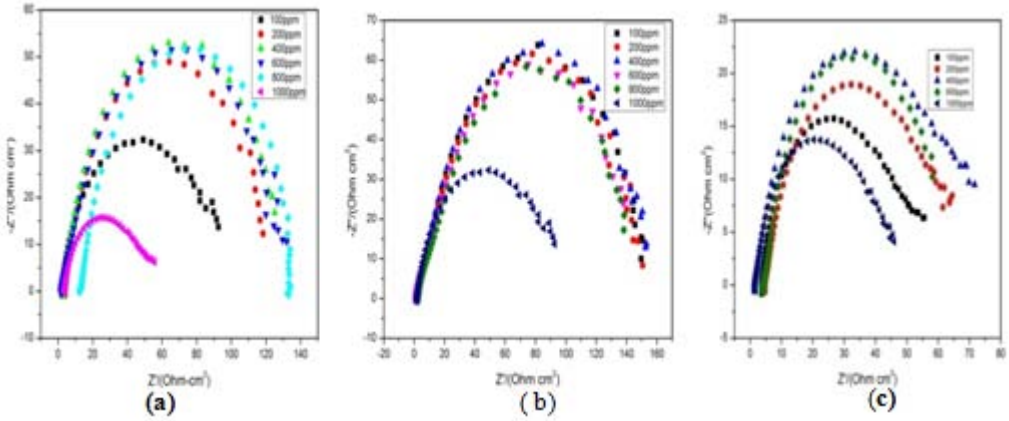

Figure 5: Nyquist plots obtained for mild steel in 1M HCl in the presence of different concentrations of [a] POT. [b] PMT. [c] PPT

Table 3: Impedance parameters for mild steel in 1M HCl in the presence and absence of different concentrations of isomers of polytoluidine

Inhibitor Conc ppm	*POT*				*PMT*				*PPT*			
	R_{ct} ($\Omega.cm^2$)	*CPE X 10^{-4} (Farad)*	*IE %*	*θ (surface coverage)*	*R_{ct} ($\Omega.cm^2$)*	*CPE X 10^{-4} (Farad)*	*IE %*	*θ (surface coverage)*	*R_{ct} ($\Omega.cm^2$)*	*CPE X 10^{-4} (Farad)*	*IE %*	*Θ (surface coverage)*
Blank	30.44	9.67	-		30.44	9.669	-		30.443	9.669	-	
100	117.99	5.68	74	0.413	140.32	6.225	79.5	0.356	141.90	6.311	78	0.347
200	120.73	5.67	74.7	0.414	143.86	6.161	79.88	0.363	143.86	5.916	78.4	0.388
400	129.33	3.21	76	0.434	263.95	6.007	88.46	0.379	144.64	5.012	78.8	0.482
600	130.31	2.91	76.2	0.699	149.86	6.916	78.8	0.398	138.06	6.0106	75.1	0.378

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1000	90.41	4.14	66	0.572	138.3	7.393	60.78	0.235	120.63	6.61	60.8	0.316

4. Conclusion

The isomers of polytoluidine were successfully synthesized by chemical oxidative polymerization method and characterized by different spectroscopic techniques. UV-VIS-NIS and FT-IR suggests the formation of quinoid and benzenoid rings which confirmed the formation of polymer.POT is found to be thermally more stable [630^{0}C] than that of PMT [480^{0}C] and PPT [430^{0}C].The polytoluidines are found to be semi conductive in nature and their conductivity is found to increase with increase in temperature. Based on the corrosion behavior investigation, the percentage inhibition efficiency of the polymers obtained from potentiodynamic polarization, and EIS measurements are found to be in good agreement and the corrosion inhibition efficiencies are in the order PMT > PPT > POT. Polarization curves demonstrated that the examined polymers behave as mixed type inhibitors. In the isomers of polytoluidine, the uniform increase in inhibition efficiency as the function of concentration deals with the adsorption phenomenon. The adsorption of inhibitors on the surface of MS is indicated by decrease in the double layer capacitance. The inhibition is due to the adsorption of the inhibitors on the steel surface and a resultant blocking of active sites. In addition, the π-electrons conjugation, quaternary nitrogen atom, and large size of the polymers also facilitate its strong adsorption on MS which leads to an efficient protection to the metal in highly corrosive medium. The results of this study clearly ascertain that isomers of polytoluidine could be considered as a candidate for the protection of MS against corrosion in an acid environment. The inhibition efficiency is found to be moderate even in the lower concentrations of inhibitors and the percentage of inhibition efficiency will considerably increase with increase in the concentration of the inhibitors from µg to mg level.

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