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Research Article

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SYNTHESIS AND CHARACTERIZATION OF POLYTHIOPHENE-ANTHRAQUINONE SULPHONIC ACID (PTH-AQSA) NANOPOWDER ON CORROSION INHIBITION OF MILD STEEL IN HCL SOLUTION

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A bstract

A new and effective polymeric corrosion inhibitor, Polythiophene-Anthraquinone sulphonic acid PTh-AQSA has been prepared and characterized by UV-Visible and FTIR spectroscopy studies. Its influence on corrosion inhibition of mild steel in 1N HCl solution was studied using chemical and electrochemical techniques. It was found that the inhibition efficiency increased with the increase of PTh-AQSA upto 700ppm and beyond this concentration, its inhibition efficiency was decreased from 95.2 to 93.1%. It indicates that 700ppm is the optimum concentration to get maximum corrosion protection for mild steel 1N HCl solution. The results obtained from chemical and electrochemical measurements are in reasonably good agreement. The potentiodynamic polarization studies revealed that PTh-AQSA act as mixed type inhibitor. Adsorption of PTh-AQSA on mild steel surface follows Langmuir adsorption isotherm. The surface characteristics of the inhibited and uninhibited mild steel were investigated by scanning electron microscope studies.

Keywords: Corrosion inhibitors, Mild steel corrosion, Polythiophene-Anthraquinone sulphonic acid, Langmuir adsorption isotherm, mixed type inhibitors.

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INTRODUCTION

Acid solutions are widely used in industries for picking, acid cleaning of boilers, descaling and oil-well acidizing etc. to control the rate of undesirable base metal corrosion, corrosion inhibitors are employed [1]. Research activities over a century have brought tremendous advances in the field of corrosion inhibitors. Hot acid solutions are generally used for removing mill scales from the metal surfaces in various industries at elevated temperatures such as 60°C in HCl solution. To minimize the percentage metal loss during this process, various compounds such as acetylenic alcohols, indoles, thiourea derivatives, dithiazones etc. are widely used [2-5]. Among these, thiourea and its derivatives are found in commercial formulations, but because of their toxic nature their use is not safe. There is a great need to find a non-toxic replacement that is compatible with

current industrial technologies. In the last two decades, there has been an increase in the use of polymeric compounds as corrosion inhibitors. Intrinsically, conducting polymers were tried as corrosion inhibitors [6].

Conducting polymers should offer better corrosion inhibition efficiency than simple organic compounds due to their inherent polar nature. The conducting polymers are rich in electrons due to the presence of long $\pi\text{-}$ electron conjugations and hence, can effectively adsorb on the metallic surfaces [7]. The literature reveals that a wide range of polymeric compounds have been successfully investigated as potential inhibitors for the corrosion of metals in aggressive media [8]. In continuation of our guest for development of corrosion inhibitors with high inhibition efficiency, the present investigation aims at the utilization of PTh-AQSA as corrosion inhibitor for carbon steel in 1N HCl solution and its corrosion inhibition performance has been studied using chemical and electrochemical methods and their results are discussed.

MATERIAL AND METHODS:-

2.1 Material Preparation

Carbon steel strips of size $4.5 \,\mathrm{cm} \times 2 \,\mathrm{cm} \times 0.2 \,\mathrm{cm}$ containing 0.14% C, 0.35% Mn, 0.17% Si, 0.025% S, 0.03% P and the remainder Fe were used for weight loss and gasometric methods. For electrochemical studies, carbon steel strips of the same composition coated with lacquer with an exposed area of $1 \,\mathrm{cm}^2$ were used. Mild steel strips were polished mechanically with emery papers of 1/0 to 4/0 grades. They were subsequently degreased with trichloroethylene before use. Analytical reagent grade HCl (Merck) and double distilled water were used for preparing test solutions for all experiments.

2.2 Synthesis of PTh-AQSA Nanopowder

Polythiophene-Cirtic acid acid (PTh- AQSA) nanopowder was prepared by cationic surfactant assisted dilute polymerization method. In this method, thiophene monomer (0.1mol) was added drop by drop into 20 mL of chloroform containing 0.014 mol of surfactant (CTAB) and 0.4 mol of anhydrous FeCl₃ and 0.1 mol of anthraquinone sulphonic acid under stirred condition under stirred condition. The ratio of

[monomer] / [surfactant] was kept about 7/1 [9]. The polymerization process was identified by changing the colour of reaction mixture into brown. The polymerization was allowed to continuous stirring for 24 hr at 30°C. The dark-brown PTh-AQSA nanopowder was collected by filtration of reaction mixture using distilled water with methanol until colourless filtrate was obtained. The PTh-AQSA powder was dried under a vacuum oven at 80°C for 6 hr. The synthesis of PTh-AQSA as shown in Figure 1.

Figure 1 Synthesis of PTh- AQSA nanopowder

The prepared PTh-AQSA powder was confirmed by FTIR spectral studies and its particles size was measured by SEM studies and it was found to be in the range of 45-50 nm (Figure 2).

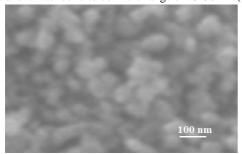


Figure 2 SEM image of PTh-AQSA Nanopowder 2.3 Corrosion Inhibitor Studies

Weight loss and gasometric measurements were carried out as described elsewhere [10, 11]. Inhibition efficiencies for different concentrations of PTh-AQSA were calculated in the absence and presence of the inhibitor in 1N HCl solution at 30°C.

To know the stability of inhibitors for a longer duration, the effect of immersion time on corrosion of carbon steel in 1N HCl was also carried out at various immersion time from 3 to 24 hours at an optimum concentration of PTh-AQSA at 30°C. From the initial and final weight of the specimen, the loss in weight was calculated at various immersion times. The degree of surface coverage (θ) was calculated from the weight loss measurements and the data were tested graphically for fitting a suitable isotherm.

Potentiodynamic polarization measurements were carried out using EG &G PAR potentiostat /galvanostat (Model - 173) analyzer a universal programmer in a conventional three – electrode glass cell at a sweep rate of 1mV per second. Both anodic and cathodic polarization curves were recorded in the absence and presence of an optimum concentration of PTh-AQSA from a cathodic to an anodic potential of \pm 250 mV (vs SCE). From the polarization curves, Tafel slopes, corrosion potential and corrosion current were calculated [12].

The electrochemical AC-impendance measurements were performed using EG & G Electrochemical impedance analyzer (model - 6310) with M38 software as described earlier [11]. A plot of Z' vs Z" were made. From the plots, the charge transfer resistance (R_t) were calculated and the double layer capacitance were then calculated [13]. The experiments were

carried out in the absence and presence of an optimum concentration of PTh-AQSA.

The effect of temperature on the corrosion behaviour of carbon steel was studied by weight loss measurements at various temperatures ranging from 30 to 90°C in the absence and presence of an optimum concentration of PTh-AQSA in 1N HCl solution for an immersion period of 3 hours.

The hydrogen permeation study was carried out using an adaptation of the modified two compartment cell described earlier [11]. The permeation current was measured in 1N HCl medium in the absence and presence of an optimum concentration of PTh-AQSA.

The carbon steel specimens were immersed in 1N HCl in the absence and presence of an optimum concentration of the inhibitor for 3h at 30°C. After 3h, the specimens were taken out, dried and kept in a desiccator. The protective film formed on the surface of carbon steel was confirmed by SEM studies with the magnification of 1000X.

III. RESULTS AND DISCUSSION

3.1 Weight loss method

The weight loss studies were carried out with various concentrations of PTh-AQSA ranging from 100 - 900 ppm to study the effect of concentration of the inhibitor and immersion time on the corrosion inhibition of PTh-AQSA in 1N HCl at 30°C for a period of 3 hours. The corrosion parameters obtained from weight loss measurements for carbon steel in 1N HCl containing various concentrations of PTh-AQSA are listed in Table -1. It was found that with increase in concentration of the PTh-AQSA from 100 to 700 ppm, the weight loss decreased and hence the inhibition efficiency increased from 76.6% to 95.2%. Beyond this concentration (700 ppm) inhibition efficiency decreased from 95.2 to 93.1%. It confirms that the optimum concentration for maximum inhibition efficiency was found to be 700 ppm. The influence of various concentrations of the PTh-AQSA on inhibition efficiency of carbon steel in 1N HCl as shown in Figure- 3.

The effect of immersion time from 3 hours to 24 hours was also studied as shown in Table- 2. The inhibition efficiency decreased from 95.2.7% to 93.3%. The effect of immersion time on percentage inhibition efficiency of carbon steel in 1N HCl at 30°C in presence of an optimum concentration 700 ppm of PTh-AQSA as shown in Figure 4. Though 93.3% inhibition efficiency was obtained even at 24hours of immersion time, the maximum inhibition efficiency was found up to 6 hrs.

Table -1. Corrosion parameters obtained from weight loss measurements for carbon steel in 1N HCl containing various concentrations of PTh-AQSA.

Conc. of PTh- AQSA (ppm)	Weight Loss (gm)	Corrosion Rate (mmpy)	Inhibition Efficiency (%)	Surface Coverage (θ)
Blank	0.2860	118.16		
100	0.0669	27.63	76.6	0.77

1					
	300	0.0446	18.42	84.4	0.84
	500	0.0283	11.69	90.1	0.90
	700	0.0136	5.61	95.2	0.95
	900	0.0197	7.84	93.1	0.93

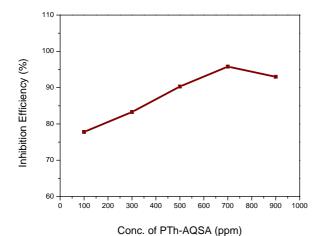


Figure.-3. Variation of inhibition efficiency with concentrations of PTh-AQSA on carbon steel in 1N HCl solution

Table -2 Effect of immersion time on percentage inhibition efficiency of carbon steel in 1N HCl at 30°C in the presence of an optimum concentration of PTh- AQSA.

			Inhib	oition I	Efficie	ncy (%))	
System	Time (h)							
	3	6	9	12	15	18	21	24
700 ppm of PTh- AQSA	95. 2	95. 1	94. 9	94. 7	94. 4	94.0	93. 6	93. 3

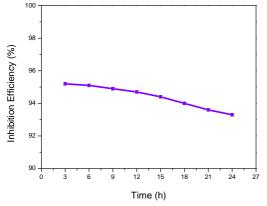


Figure- 4 Effect of immersion time on percentage inhibition efficiency of carbon steel in 1N HCl at 30°C in presence of an optimum concentration (700 ppm) of PTh- AQSA.

3.2 Gasometric method

Table-3 shows in the gasometric method, the increase in concentrations of PTh-AQSA from 100 to 700 ppm, decreased the volume of hydrogen gas evolved was found to decrease from 1.6 to 0.5 mL and hence the inhibition efficiency increased from 75.0 to 88.9%. Beyond this concentration, the inhibition efficiency decreased from 77.8 to 93.0% as shown in Table 4. Hence, the optimum concentration of PTh-AQSA was found to be 700 ppm. The volume of gas evolved during the corrosion of carbon steel in 1N HCl in the absence and presence of various concentrations of PTh-AQSA as shown in Figure 5. It could be observed that PTh-AQSA has better ability to inhibit the corrosion of carbon steel in acid solution.

Table- 3 Inhibition efficiency obtained from gasometric measurements for carbon steel in 1N HCl containing various concentrations of PTh-AQSA at 30° C.

Conc. of PTh- AQSA	Volume of Hydrogen Gas Evolved	Inhibition Efficiency (%)	
(ppm)	(mL)	(70)	
Blank	7.2		
100	1.6	77.8	
300	1.2	83.3	
500	0.7	90.3	
700	0.3	95.8	
900	0.5	93.0	

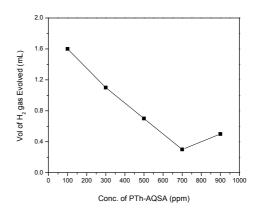


Figure- 5 Variation of volume of gas evolved for the corrosion of carbon steel in 1N HCl of various concentration of PTh-AQSA.

3.3 Potentiodynamic Polarization Method

The polarization curves for carbon steel in 1N HCl with various concentrations of PTh- AQSA as shown in Figure 6. It can be seen that the corrosion potential was not shifted significantly in presence of PTh- AQSA control both anodic and cathodic reactions to inhibit the corrosion of carbon steel by blocking active sites on the carbon steel surface [14].

The potentiodynamic polarization parameters for carbon steel in 1N HCl in the absence and presence of an optimum concentration of PTh-AQSA are given in Table-4. It can be seen that in the presence of an optimum concentration of inhibitor, the corrosion current decreased from 3.68 μA /cm² to 0.17 μA /cm². The extent of its decrease increased with PTh-AQSA concentration from 100 to 700 ppm. This behaviour reflected the ability of PTh-AQSA to inhibit the corrosion of carbon steel in 1N HCl. It was observed that with increase in concentration of PTh- AQSA from 100 to 700 ppm, the maximum inhibition efficiency of 95.8% was observed.

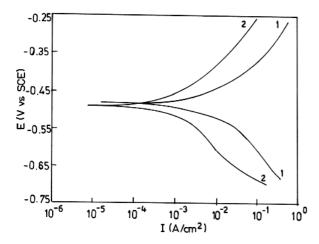


Figure- 6 Potentiodynamic polarization curves for carbon steel in 1N HCl solution in the absence and presence of an optimum conc. of PTh- AQSA.

Table -4 Potentiodynamic polarization parameters for carbon steel in 1N HCl in the absence and presence of an optimum concentration of PTh-AQSA.

Conc. of PTh- AQSA	E _{corr}	I _{corr}	Tafel Slope (mV/decade)		Inhibiti on Efficien cy
(ppm)	SCE)	(μA/c m ²)	b _a	b _c	(%)
Blank	-488	3.68	84.4	102. 2	
700	- 490	0.17	84.1	102. 4	95.4

3.4 AC - impedance measurements

The corrosion behaviour of carbon steel in 1N HCl in the absence and presence of PTh-AQSA was investigated by AC-impedance measurement method to evaluate the charge transfer resistance (R_t) and double layer capacitance (C_{dl}). Figure 7 shows the impedance diagrams for carbon steel in 1N HCl in the absence and presence of an optimum concentration of PTh-AQSA and their corresponding impedance parameters from these investigations are

given in Table 5. It can be seen from the Nyquist plots that the curve are almost of semicircular nature and is due to the charger - transfer process, which mainly controls the corrosion of carbon steel in 1N HCl solution [6]. Deviations of perfect circular shape are often referred to the frequency dispersion of interfacial impedance. This anomalous phenomenon may be attributed to the in homogeneity of the electrode surface arising from surface roughness or interfacial phenomena [15].

In the presence of PTh-AQSA, the value of R_t increased and the value of C_{dl} decreased. The decrease in C_{dl} showed that the adsorption of the inhibitor took place on the carbon steel surface in acid solution. The increase in the value of R_t led to increase in the inhibition efficiency. The maximum R_t value of 458.6 Ω cm² and the minimum C_{dl} value of 520 $\mu F/cm^2$ was obtained at an optimum concentration of 700 ppm of PTh-AQSA gave the maximum inhibition efficiency of 95.5%. This result has good agreement with the results obtained from non-electrochemical methods.

Table - 5 Impedance parameters for the corrosion of carbon steel in 1N HCl in the absence and presence of an optimum concentration of PTh- AQSA at $30^{0}C$

Conc. of PTh- AQSA (ppm)	R _t (Ω cm ²)	C _{dl} (µF/cm ²)	Inhibition Efficiency (%)
	20.2	5046	
700	458.6	520	95.5

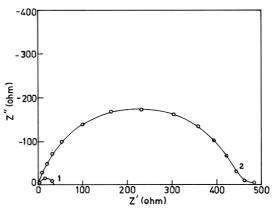


Figure -7 Impedance diagrams for carbon steel in 1N HCl solution in the absence and presence of an optimum concentration of PTh- AQSA (1) Blank (2) 700 ppm of PTh- AQSA

3.5 Effect of Temperature

The effect of temperature in the range 30°C to 90°C on the corrosion behaviour of carbon steel in 1N HCl in the absence and presence of the PTh- AQSA was studied using weight loss

method is shown in Table-6 (Figure 8). It can be seen from the table that the increase in corrosion rate was more pronounced with the rise in temperature for the uninhibited acid solution than the inhibited solution. The PTh-AQSA would have adsorbed on the carbon steel surface at all temperatures studied. As temperature increased from 30°C to 90°C, the inhibition efficiency was found to decrease from 95.2% to 88.5%. This suggests that the adsorption of the PTh- AQSA on the carbon steel may be due to physisorption.

Table -6 Corrosion of carbon steel in 1N HCl at different temperatures obtained by weight loss method in the absence and presence of an optimum concentration of 700 ppm of PTh-AQSA.

System	System Temperature (°C)		Inhibition Efficiency (%)	
	30	118.16		
Blank	50	147.91		
	70	186.21		
	90	239.90		
	30	5.67	95.2	
700 ppm of PTh- AQSA	50	9.77	93.6	
	70	16.22	91.3	
	90	27.54	88.5	

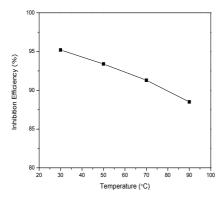


Figure- 8 Effect of temperature on the corrosion inhibition efficiency of carbon steel in 1N HCl in presence of an optimum concentration (700ppm) of PTh-AQSA.

3.6 Mechanism for the corrosion inhibition of PTh- AQSA

The Arrehenius plot for carbon steel immersed in 1N HCl solution in the absence and presence of an optimum concentration (700 ppm) of PTh- AQSA as given in Figure 9. The calculated values of activation energy (Ea), enthalpy of adsorption (Δ H), free energy of adsorption (Δ G $^{\circ}$) and entropy of adsorption

(ΔS) are shown in Table-7. The activation energy (E_a) was found to be 22.14 KJ mol⁻¹ for 1N HCl and increased to 27.40 KJ mol⁻¹ in the presence of PTh- AQSA suggesting that the adsorbed organic matter formed a physical barrier to charge and mass transfers, leading to reduction in corrosion rate. The higher value of E_a in presence of the inhibitor compared to that in the absence of the inhibitor was attributed to physisorption [16,17].

The negative sign of free energy of adsorption indicates that the adsorption of PTh- AQSA on carbon steel surface as a spontaneous process. The ΔG° values are in the range -18.54 KJ mol $^{-1}$ to -19.36 KJ mol $^{-1}$. As the values of free energy of adsorption are less than -20 KJ mol $^{-1}$, the mode of adsorption is physisorption. The positive values of enthalpy of adsorption (ΔH) shows that the reaction is endothermic and the adsorption of the inhibitor on the metal surface took place. Entropy of adsorption (ΔS) remained a positive value and this reflects the formation of an ordered stable layer of the inhibitor molecule on the surface. The positive value of entropy indicates that the reaction was spontaneous and feasible.

Figure- 9 Arrehenius plots for carbon steel immersed in 1N HCl solution in the absence and presence of an optimum concentration of PTh- AQSA (a) Blank (b)700 ppm of PTh-AQSA.

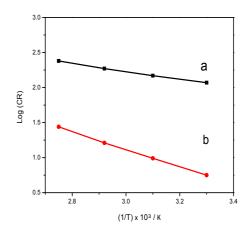


Table- 7 Calculated values of activation energy (Ea), enthalpy of adsorption (ΔH), free energy of adsorption (ΔG°) and entropy of adsorption (ΔS) for carbon steel in the absence and presence of an optimum concentration 700 ppm of PTh- AQSA in 1N HCl solution.

System	Temp. (K)	E _a (KJ mol ⁻ 1)	ΔG° (KJ mol ⁻ 1)	ΔH (KJ mol ⁻ 1)	ΔS (KJ mol ⁻¹)
	303			8.43	
Blank	323	9.14		8.26	
	343	10.61		8.10	

	363	13.11		7.93	
	303		- 18.54	21.78	0.1331
700 ppm of	323	22.14	18.86	21.61	0.1253
PTh- ASQA	343	23.35	- 19.17	21.45	0.1184
	363	27.40	- 19.36	21.28	0.1119

The polymeric inhibitor, PTh-AQSA could adsorb on the metal surface via the lone pair of electrons present on their nitrogen atoms. The inhibitor might have adsorbed on the surface of the carbon steel and formed a compact protective thin layer on the surface of carbon steel. The corrosion of carbon steel in HCl solution is heterogonous one, composed of anodic and cathodic reactions. The adsorption of PTh-AQSA molecules on the carbon steel surface makes a barrier for mass and charge transfers. This situation leads to a protection of carbon steel surface from the attack of the aggressive ions of acid solution. The degree of protection increased with increasing of the surface fraction occupied by the adsorbed molecules. As the inhibitor concentration increased, the number of adsorbed molecules on the surface increases.

Surface coverage (θ) was calculated from the inhibition efficiency values using weight loss method, and was used to represent the fraction of the surface occupied by the adsorbed molecules. The values of surface coverage (θ) for various concentrations of the inhibitor are given as Table-1. The use of adsorption isotherms provided useful insight into the corrosion inhibition mechanism. A plot of C/θ versus C gave a straight line with unit slope suggests that the adsorption of various concentrations of PTh- AQSA on the surface of carbon steel in 1N HCl followed Langmuir adsorption isotherm (Figure- 10).

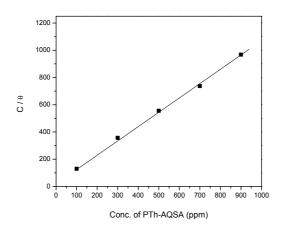


Figure -10 Langmuir adsorption isotherm plot for the adsorption of various conc. of PTh- AQSA on the surface of carbon steel in 1N HCl solution.

3.7 Surface Analysis

The carbon steel specimens, after immersion in 1N HCl solution for 3 hours at 30°C in the absence and presence of an optimum concentration 700 ppm of the PTh-AQSA, were taken out, dried and kept in a dessicator. Surface examination revealed the presence of a film as shown in Figure- 11 (a & b).

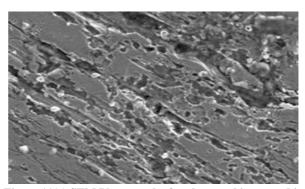


Figure 11(a) SEM Photograph of carbon steel immersed in 1N HCl solution (blank).

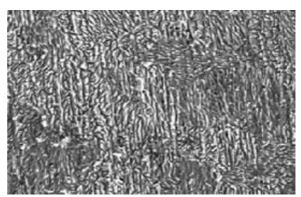


Figure 11(b) SEM Photograph of carbon steel immersed in 1N HCl solution containing an optimum concentration (700 ppm) of PTh- AQSA.

Conclusions:-

PTh-CA nano powder was prepared and characterized. It inhibits the corrosion of carbon steel in 1N HCl solution at a concentration of 700 ppm. PTh-AQSA control both anodic and cathodic reactions by blocking the active sites of steel surface and is inhibitor of mixed type. The adsorption of PTh-AQSA on carbon steel surface in 1N HCl solution is found to obey Langmuir adsorption isotherm.

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