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Corrosion Inhibition and Adsorption Studies of some Barbiturates on Mild Steel/Acid interface

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Four new corrosion inhibitors namely 5-(3-phenylallylidene) pyrimidine-2,4,6-trione (PPT), 5-(2-hydroxybenzylidene) pyrimidine-2,4,6-trione (HPT), 5-benzlidenepyrimidine-2,4,6-trione (BPT) and 5-(4-nitrobenzylidene) pyrimidine-2,4,6-trione (NPT) were synthesized and their inhibiting effect on corrosion of mild steel in 1M HCl was investigated using electrochemical impedance spectroscopy (EIS), Tafel polarization and weight loss measurements. The adsorption of the inhibitors obeyed Langmuir isotherm. Both thermodynamic and activation parameters were calculated and discussed. Polarization curves indicate that the inhibitors are of mixed type.

Keywords: Acid corrosion; Mild steel; EIS; Tafel polarization; Inhibitor; Langmuir isotherm

1. INTRODUCTION

Mineral acids, particularly hydrochloric acid are frequently used in industry during cleaning, acid pickling, acid descaling and oil well acidizing [1-3]. Due to aggressiveness of acids, inhibitors are often used to reduce the rate of dissolution of metals. Organic compounds containing heteroatom's, polar functional groups and π-electrons as active centers have been reported as effective corrosion inhibitors [4-13].

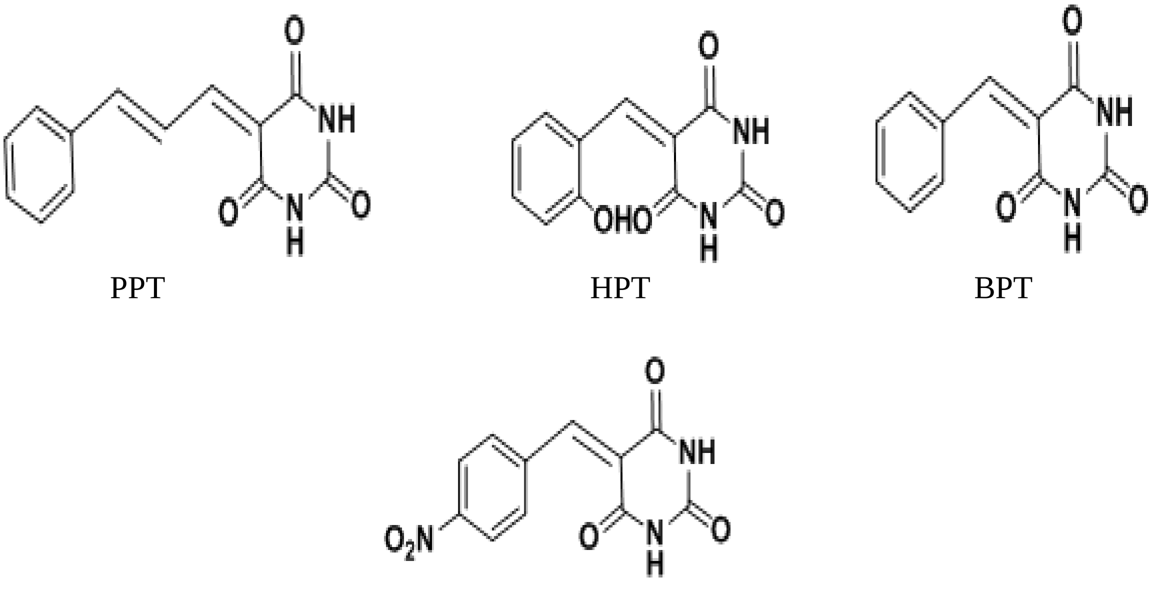
Barbituric acid and its derivatives exhibit a wide range of biological activities such as antibacterial and anti-hypertensive agents [14]. In continuation of our work on the development of heterocyclic compounds as corrosion inhibitors in acidic environment [8], we report here the inhibiting effect of four Arylidene barbiturates viz. 5-(3-phenylallylidene) pyrimidine-2,4,6-trione (PPT), 5-(2-hydroxybenzylidene) pyrimidine-2,4,6-trione (HPT), 5-benzlidenepyrimidine-2,4,6-trione (BPT) and

5-(4-nitrobenzylidene) pyrimidine-2,4,6-trione (NPT) on the corrosion of mild steel in 1 M HCl using polarization measurements, impedance techniques and weight loss measurements.

2. EXPERIMENTAL

2.1. Materials used

Four Arylidene barbiturates were synthesized according to the literature [15]. IUPAC names, Abbreviated names and chemical structure of the synthesized Arylidene barbiturates are given in Fig. 1.



PPT HPT BPT

NPT

Figure 1. Molecular structure of Inhibitors

The stock solutions of the synthesized compounds were prepared in 1 M HCl solution. The corrosion tests were performed on mild steel specimens with a composition (wt. %) C: 0.076, P: 0.012, Si: 0.026, Mn: 0.192, Cr: 0.050, Cu: 0.135, Al: 0.023, Ni: 0.050 and Fe balance. The mild steel specimens with dimensions 2.5 cm × 2.0 cm × 0.025 cm were used for weight loss measurements. For electrochemical measurements, mild steel specimens of 7.5 cm long stem with exposed surface area of 1.0 cm^2 (rest being coated with commercially available lacquer) were used. The surface pretreatment was carried out by polishing with 600, 800, 1000 and 1200 grit emery paper, followed by washing with double distilled water and finally degreased with acetone and dried at room temperature. All chemicals used were of analytical reagent grade and were used without further purification. The solutions were prepared using double distilled water and all experiments were carried out in un-stirred solutions.

2.2. Methods

2.2.1. Electrochemical measurements

All the electrochemical experiments were performed using a three electrode assembly connected to Gamry Instrument Potentiostat/Galvanostat with a Gamry framework system based on ESA 400. Gamry applications include EIS 300 for EIS measurements and Echem Analyst (version 5.50) software package for data fitting. A standard calomel electrode (SCE) was used as reference electrode, platinum electrode was used as an auxiliary electrode and mild steel of 1 cm^2 was the working electrode. All potentials were measured versus SCE.

EIS measurements were carried out in a frequency range from 10^5 Hz to 10^{-2} Hz under potentiostatic conditions, with amplitude of 10 mV peak-to-peak, using AC signal at open circuit potential (E_{OC}). LPR experiments were performed from -20 to +20 mV versus E_{OC} at a scan rate of 0.125 mV s^{-1}.

Potentiodynamic polarization curves were obtained by changing the electrode potential automatically from 250 to +250 mV versus E_{OC} at a scan rate of 1 mVs^{-1}. All the experiments were performed after an immersion time of 30 min in 1 M HCl in the absence and presence of inhibitors.

2.2.2. Weight loss measurements

The weight loss study was done on mild steel strips of dimension 2.5 cm × 2.0 cm × 0.025 cm sizes. Weight loss study was carried out at 308 K for 3 h time duration in 1 M HCl solution. The inhibition efficiency η% and surface coverage (θ) were determined using following equation:

$$\eta\% = \frac{C_{\mathrm{R}} - C_{\mathrm{R(i)}}}{C_{\mathrm{R}}} \times 100 \qquad (1)$$

$$\theta = \frac{C_{\mathrm{R}} - C_{\mathrm{R(i)}}}{C_{\mathrm{R}}} \qquad (2)$$

where C_R and $C_{R(i)}$ are the values of the corrosion rates (mg cm^{-2} h^{-1}) of mild steel in absence and presence of inhibitors, respectively.

3. RESULTS AND DISCUSSION

3.1. Gravimetric measurements

3.1.1. Effect of inhibitor concentration

The values of percentage inhibition efficiency (η%) and corrosion rate (CR) obtained from weight loss method at optimum concentration of inhibitors in 1 M HCl at 308K are summarized in Table 1.

Table 1. Corrosion parameters obtained from gravimetric measurement for mild steel in1M HCl in absence and presence of optimum (350 mg/L) concentrations of inhibitors at 308 K

C_{inh} (mg/L)	C_R (mm/year)	θ	η (%)
Blank	77.9	-	-
PPT	2.22	0.971	97.1
HPT	3.71	0.952	95.2
BPT	6.30	0.919	91.9
NPT	9.27	0.880	88.0

It is evident from Table 1 that the corrosion rate decreases as the concentration of inhibitor increases.

3.1.2. Effect of temperature

Table 2. Corrosion parameters obtained from gravimetric measurement for mild steel in 1M HCl in presence of optimum concentration (350 mg/ L) of inhibitors at different temperatures

Inhibitors	**Temperature (K)**	**C_R (mm/year)**	**θ**	**η (%)**
Blank	308	77.9	--	--
	318	107.6	--	--
	328	162.5	--	--
	338	208.5	--	--
PPT	308	2.22	0.971	97.1
	318	8.53	0.920	92.0
	328	23.74	0.853	85.3
	338	70.49	0.661	66.1
HPT	308	3.71	0.952	95.2
	318	11.13	0.896	89.6
	328	28.56	0.824	82.4
	338	77.91	0.626	62.6
BPT	308	6.30	0.919	91.9
	318	16.69	0.844	84.4
	328	37.10	0.771	77.1
	338	83.48	0.599	59.9
NPT	308	9.27	0.880	88.0
	318	20.40	0.810	81.0
	328	47.86	0.705	70.5
	338	97.58	0.532	53.2

To evaluate the corrosion process of mild steel in 1M HCl, weight loss measurements were carried out in the temperature range 308–338 K in the absence and presence of optimum concentration of inhibitors in 1M HCl. The values of corrosion rate (CR) and inhibition efficiency (η%) are listed in Table 2.

The above table indicates that the η% decreases with increasing temperature. This occurs due to increase in the dissolution process of mild steel and partial desorption of the inhibitor from the metal surface.

The log of corrosion rate (CR) is a linear function with 1/T [16] (Arrhenius equation)

$$C_{\mathrm{R}} = A\exp\left(\frac{-E_{\mathrm{a}}}{RT}\right) \tag{3}$$

where E_a represents apparent activation energy, R the gas constant, A is the pre-exponential factor. A plot of log of corrosion rate (CR) (obtained by weight loss measurement) versus 1/T gave straight lines as shown in the Fig. 2 with a slope of $E_a/2.303R$.

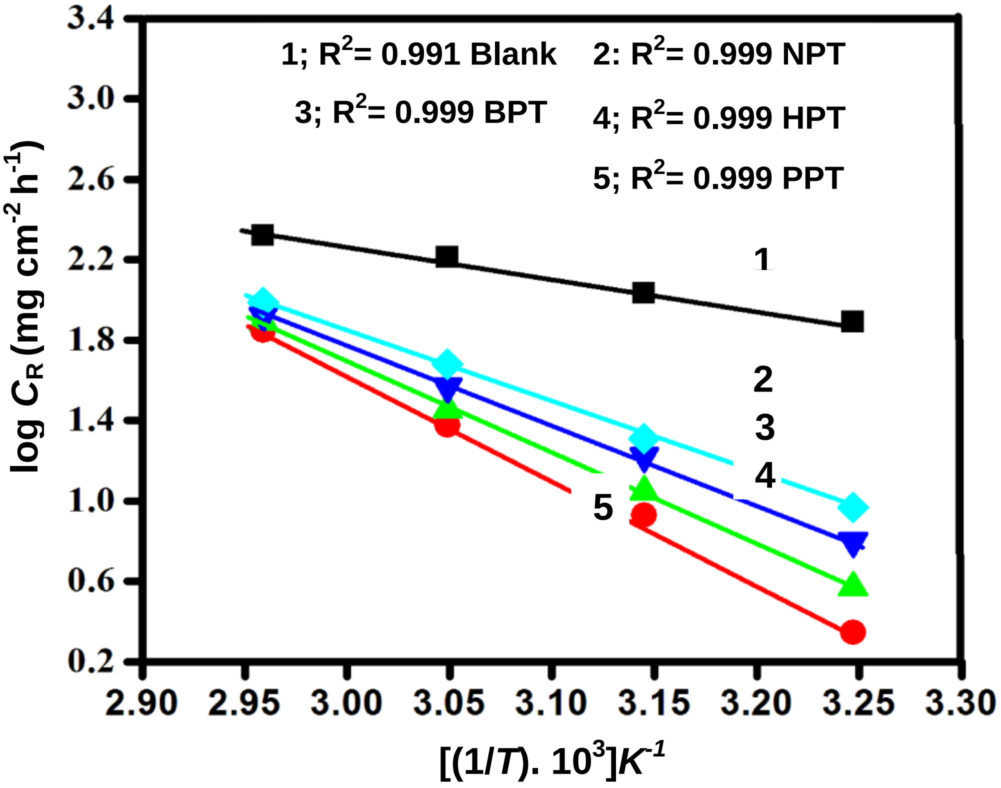


Figure 2. Arrhenius plots of mild steel in 1M HCl in absence and presence of optimum concentration of inhibitors.

The values of activation energy obtained are listed in Table 3.

Table 3. Activation parameters for mild steel dissolution in 1M HCl in the absence and presence of optimum (350 mg/ L) concentration of inhibitors

Inhibitors	E_a (kJ mol^{-1})	ΔH^* (kJ mol^{-1})	ΔS^* (J K^{-1} mol^{-1})
Blank	27.9	25.4	-147.4
PPT	98.72	96.0	73.54
HPT	87.23	84.59	40.22
BPT	74.06	71.34	1.93
NPT	68.51	65.87	-12.96

The data showed that the thermodynamic activation function (E_a) of the corrosion in mild steel in 1 M HCl solution in the presence of inhibitor are higher than those in the free acid solution suggesting physical adsoption of inhibitors on metal surface [17].

An alternative formulation of Arrhenius equation is [18]:

$$C_R = \frac{RT}{Nh}\exp\left(\frac{\Delta S_a}{R}\right)\exp\left(-\frac{H_a}{RT}\right) \quad (4)$$

where, h is Planck's constant, N is Avogadro number, ΔS_a the entropy of activation, and ΔH_a is the enthalpy of activation. A plot of log CR/T versus 1/T gave straight lines as shown in Fig. 3

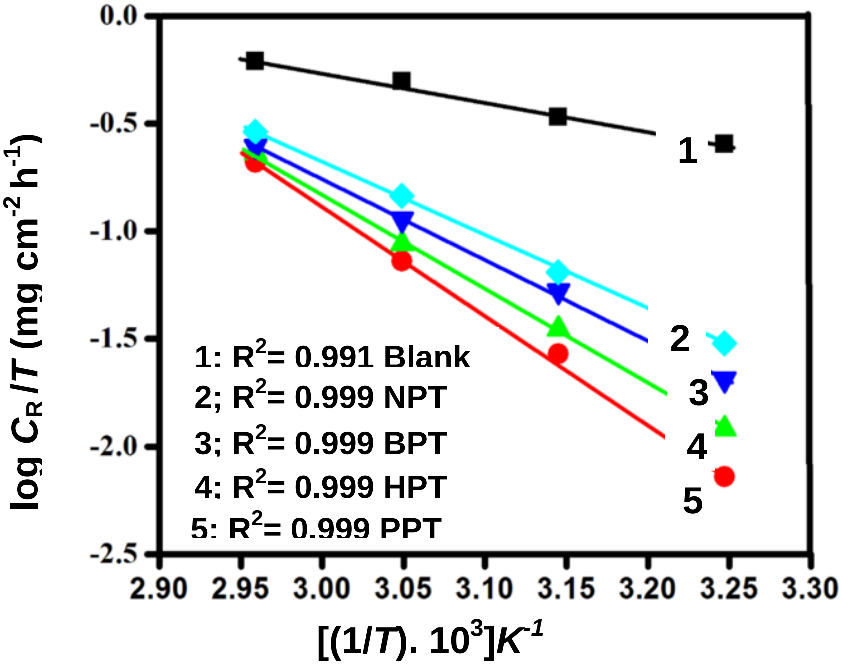


Figure 3. Transition-state plot of mild steel in 1M HCl in absence and presence of optimum concentration of inhibitor with a slope of (ΔHa/2.303R) and an intercept of [log(R/Nh) + (ΔSa/2.303R)] from which the values of ΔH_a and ΔS_a were calculated and given in Table 3.

The positive signs of activation enthalpies (ΔH_a) reflect the endothermic nature of dissolution process. The shift towards positive value of entropies (ΔS_a) implies that the activated complex in the rate determining step represents dissociation rather than association process, which means that disordering, increases on going from reactants to the activated complex. [19] Free energy of adsorption (ΔG_{ads}) and K_{ads} calculated using the following equations [20] and their value are given in Table 4.

$$\Delta G^{o}_{\mathrm{ads}} = -RT\ln(55.5K_{\mathrm{ads}}) \tag{5}$$

Where K_{ads} is given by

$$K_{ads}C = \frac{\theta}{1-\theta} \tag{6}$$

where θ is the surface coverage, C is the inhibitor concentration, K_{ads} is the equilibrium constant of adsorption process.

Table 4. Thermodynamic parameters for the adsorption of inhibitors on mild steel at optimum (350 mg/ L) in 1M HCl at different temperatures

Inhibitor	**Temperature (K)**	**K_{ads} (10^3 M^{-1})**	**$-\Delta G^{\circ}_{ads}$ (kJ mol^{-1})**
PPT	308	40.19	37.44
	318	13.80	35.83
	328	6.966	35.09
	338	2.340	33.09
HPT	308	23.80	36.10
	318	10.34	35.06
	328	5.62	34.50
	338	2.00	32.66
BPT	308	13.62	34.67
	318	6.49	33.83
	328	4.04	33.60
	338	1.79	32.34
NPT	308	8.80	33.55
	318	5.11	33.20
	328	2.86	32.67
	338	1.36	31.58

The value of $\Delta G°_{ads}$ for the inhibited system is less than -40 kJ mol^{-1} which signifies physisorption [21]. The K_{ads} values decrease with increasing temperature indicating that the desorption process is enhanced at elevated temperatures.

3.1.3. Adsorption isotherm studies

In order to understand the mechanism of corrosion inhibition, the adsorption behaviour of organic adsorbate on the mild steel surface must be known [22]. The degree of surface coverage (θ) for optimum concentration of inhibitors in 1 M HCl was evaluated from weight loss values. These data were examined graphically by fitting in various isotherms. A straight line was obtained on plotting log(θ/1- θ) versus log C (Fig. 4).

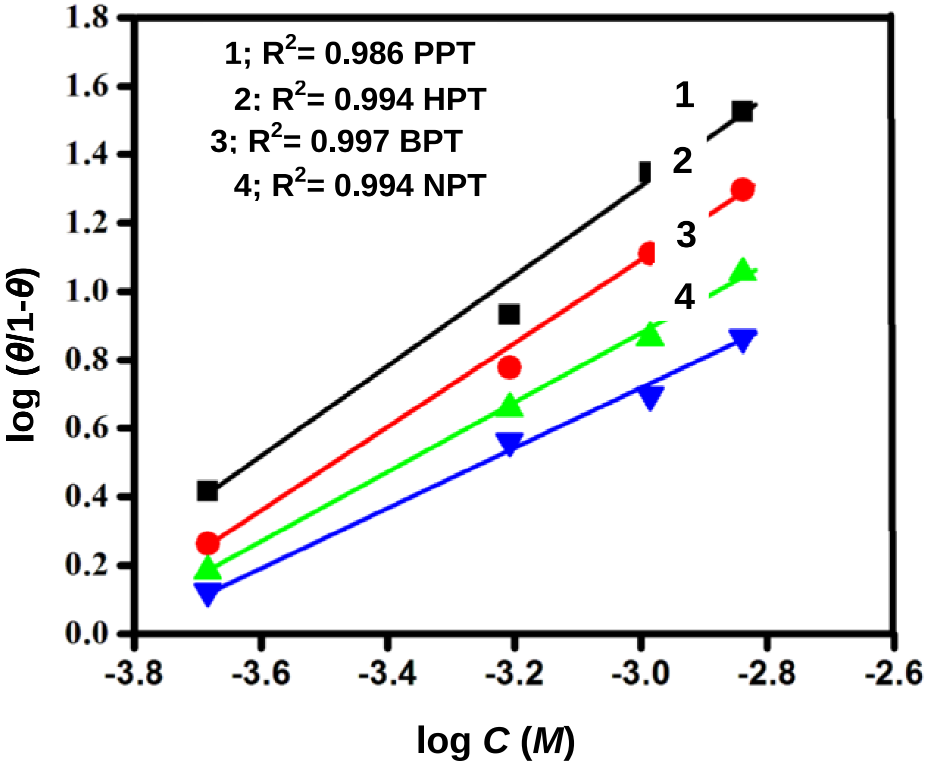


Figure 4. Langmuir's isotherm for adsorption of inhibitor on mild steel surface in 1M HCl

The straight lines suggest that the adsorption of the compound on the mild steel surface follows Langmuir's adsorption isotherm.

3.2. Electrochemical measurements

3.2.1 Tafel polarization

The potentiodynamic Tafel polarization curves of mild steel in the absence and presence of optimum concentration of Arylidene barbiturates in 1M HCl are shown in Fig.5.

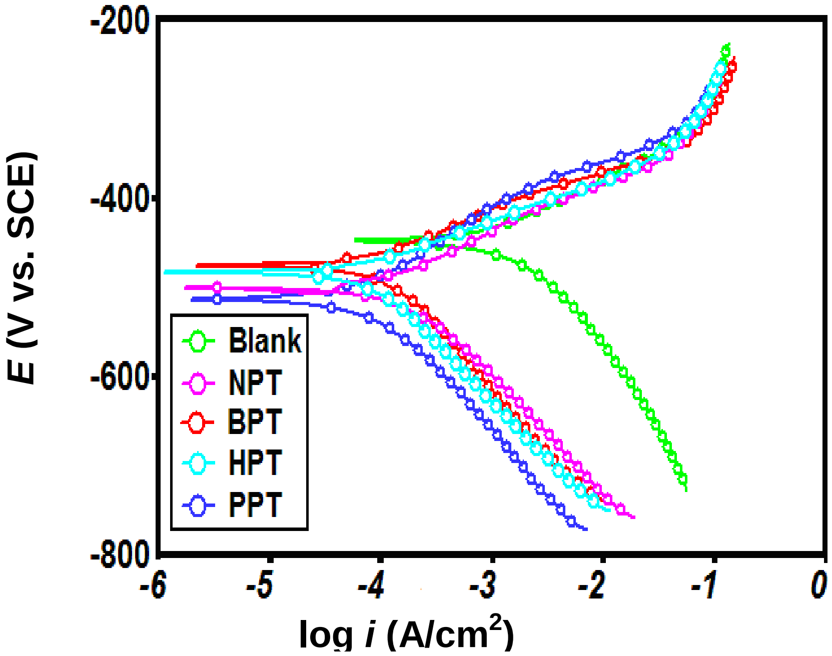


Figure 5. Potentiodynamic polarization curves for mild steel in 1M HCl in absence and presence of optimum (350 mgL^{-1}) concentrations of inhibitors.

The important corrosion parameters derived from these curves are given in Table 5.

Table 5. Electrochemical polarization parameters for mild steel in 1M HCl solution in the absence and presence of optimum (350 mgL^{-1}) concentrations of inhibitors

C_{inh} (mg/L)	E_{corr} (mV/SCE)	I_{corr} (μA/cm^2)	b_a (mV/dec)	b_c (mV/dec)	η (%)
Blank	-445	1160	71.0	114.6	–
PPT	-483	76.9	50.7	128.2	93.37
HPT	-511	87.9	101.8	135.5	92.42
BPT	-490	89.1	98.3	116.8	92.31
NPT	-474	117	60.0	134.1	89.90

The results shown in Table 5 clearly indicate that the Arylidene barbiturates bring down the corrosion current without causing any appreciable change in values of corrosion potential suggesting that they are mixed type inhibitor. Also the value of b_a and b_c did not show any significant change indicating that the studied inhibitors are mixed-type.

3.2.2. Electrochemical impedance

The impedance behavior of mild steel in 1 M HCl with and without addition of optimum concentration of Arylidene barbiturates is presented as Nyquist plot in Fig 6.

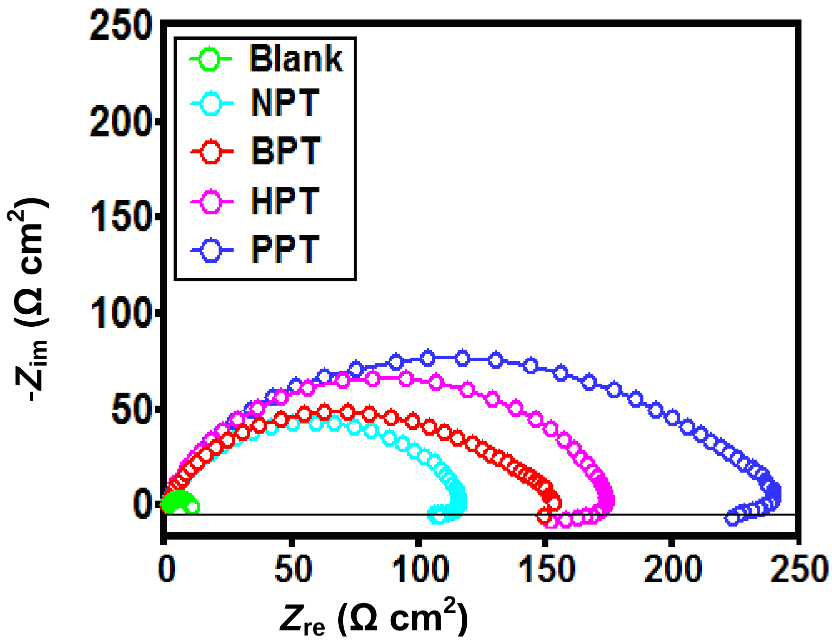


Figure 6. Nyquist plots for mild steel in 1M HCl in absence and presence of optimum concentrations of inhibitor at 308 K

All the impedance parameters were calculated from Nyquist plot . Fig. 7 and 8. were use to fit and analyze EIS data.

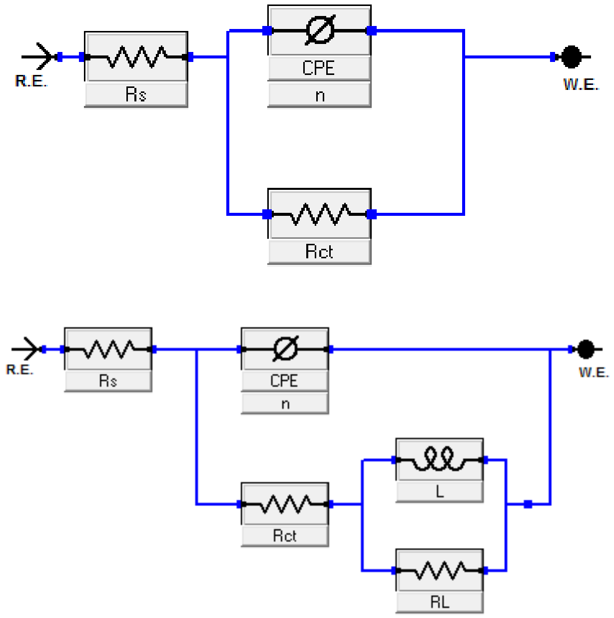


Figure 7 and 8. Equivalent circuit used to fit the EIS data

These circuits consist of R_s (the resistance of solution between working electrode and counter electrode), C_{dl} in parallel to the R_{ct}, and the R_{ct} is in series to the parallel of inductive elements L and RL. The presence of L in the impedance spectra in the presence of investigated inhibitors indicated that iron is still dissolved by the direct charge transfer at the inhibitor adsorbed electrode surface [23]. The CPE is substituted for the capacitor to fit the semicircle more accurately. The admittance, YCPE, and impedance, ZCPE, of a CPE are expressed as [24]:

$$Y_{\mathrm{CPE}} = Y_0 (j\omega)^n \tag{7}$$

and

$$Z_{\mathrm{CPE}} = \left(\frac{1}{Y_0}\right)\left[(j\omega)_n\right]^{-1} \tag{8}$$

where Y_0 is the admittance, j is the square root of -1, ω is angular frequency ($\omega = 2\pi f_{max}$) at which the imaginary part of the impedance ($-Z_{im}$) is maximal and f_{max} is AC frequency at maximum, n is the phase shift, which can be used as a gauge of the heterogeneity or roughness of the mild steel surface. The CPE can be expressed by the values of n if resistance (n = 0, Y_0 = R), capacitance (n = 1, Y_0 = C), inductance (n = -1, Y_0 = L) and Warburg impedance (n=0.5, Y0 = W) [25]. The impedence value are given in Table 6.

Table 6. Electrochemical impedance parameters for mild steel in 1M HCl solution in the absence and presence of optimum (350 mgL^{-1}) concentrations of inhibitors

C_{inh} (mgL^{-1})	R_s (Ω)	R_{ct} (Ω cm^2)	*n*	Y_0 (µF/cm^2)	C_{dl} (µF/cm^2)	L (Hcm^2)	RL (Ωcm^2)	*η* (%)
Blank	0.58	12.98	0.790	250.1	109.5	--	--	--
PPT	1.195	227.4	0.809	55.12	24.03	160.3	47.84	94.29
HPT	0.979	160.6	0.834	70.58	28.12	62.0	19.25	91.91
BPT	0.873	140.0	0.840	88.86	42.34	44.4	4.4	90.72
NPT	0.973	122.2	0.860	102.4	51.70	23.2	3.9	89.37

It is evident from the Table 6 that the addition of inhibitors increase R_{ct} values, which is attributed to the formation of protective film on the metal/ solution interface [25,26]. The maximum R_{ct} value (227.4 Ω cm^2) was obtained for PPT at 350 mg L^{-1}. The inhibition efficiency (η %) using R_{ct} values were calculated from the equation [27]:

$$\eta\% = \left(1 - \frac{R_{ct}}{R_{ct(i)}}\right) \times 100 \quad (9)$$

where $R_{ct(i)}$ and R_{ct} are the charge transfer resistance in presence and absence of Arylidene barbiturates, respectively. The double layer capacitance (C_{dl}) values were calculated using the following equation [28]:

$$C_{dl} = \frac{Y\omega^{n-1}}{\sin\left(n(\pi/2)\right)} \quad (10)$$

It is seen that C_{dl} value decreases in presence of inhibitors which is attributed to local dielectric relaxation of inhibitor molecules on metal surface or increase in thickness of electrical double layer [29]. C_{dl} is also related to the surface area according to the expression of the Helmholtz model [28]:

$$C_{\mathrm{dl}} = \frac{\varepsilon\varepsilon_0}{d} S \quad (11)$$

where ε_0 is the permittivity of free space (8.854×10^{-12} $\mathrm{Fm^{-1}}$) and ε is the local dielectric constant of medium, S is the surface area of the electrode. Equation (11) suggests that C_{dl} is inversely proportional to the thickness of protective layer d.

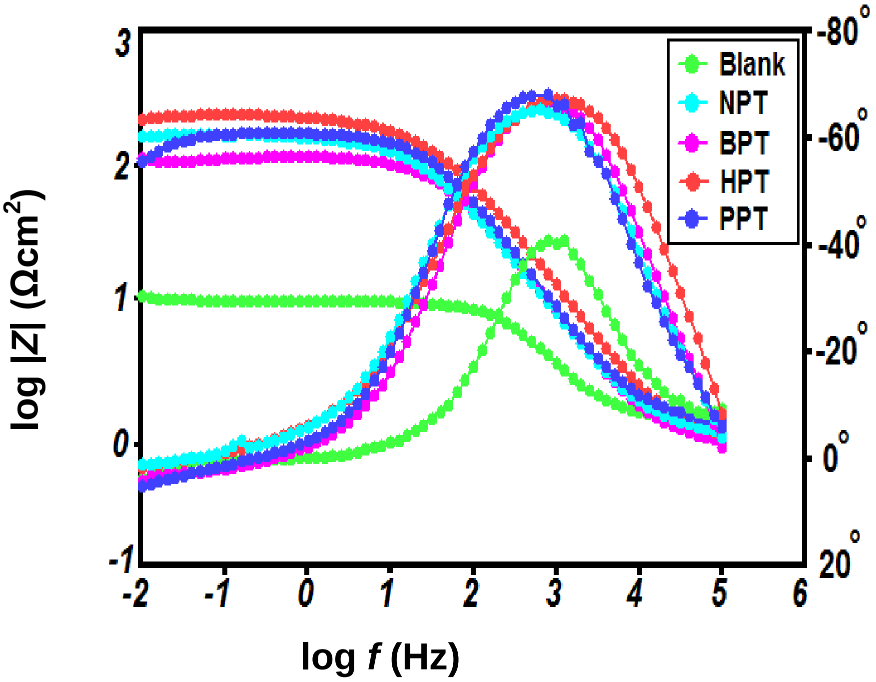


Figure 9. Bode (log *f* vs. log |*Z*|) and phase angle (log *f* vs. *α*) plots of impendence spectra for mild steel in 1M HCl in absence and presence of optimum concentration of inhibitors at 308 K.

Figure 9 depicts the Bode impedance magnitude and phase angle plots recorded for mild steel electrode immersed in 1 M HCl in the absence and presence of optimum concentration of Arylidene barbiturates at its open circuit potential.

The slope of Bode impedance magnitude plots at intermediate frequencies, S, and the maximum phase angles, α, showed deviation from the values of -1 and 90°, respectively, and is given in the Table 7.

Table 7. The slopes of the Bode impedance magnitude plots at intermediate frequencies (S) and the maximum phase angles (α) for mild steel in 1M HCl solution at optimum (350 mgL^{-1}) concentrations of inhibitors at 308 K.

C_{inh} (mgL^{-1})	$-S$	$-\alpha^{\circ}$
Blank	0.502	40.90
PPT	0.741	67.36
HPT	0.709	65.35
BPT	0.742	66.51
NPT	0.774	66.81

The Bode phase angle plots show single maximum (one time constant) at intermediate frequencies. The broadening of phase angle plots in presence of Arylidene barbiturates accounts for the formation of a protective layer on electrode surface [30].

3.3 Mechanism of corrosion inhibition

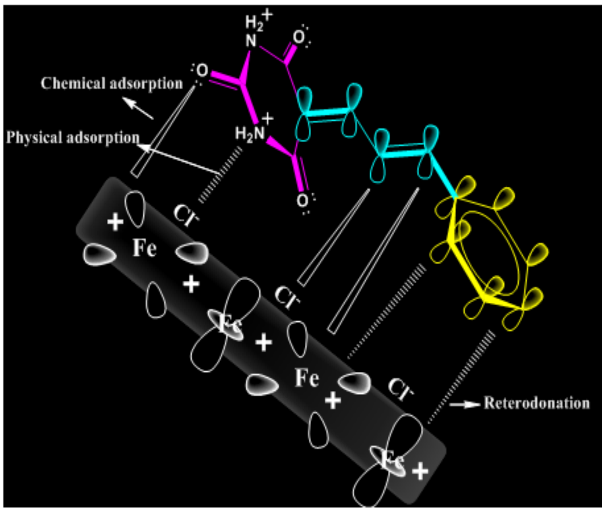


Figure 10. Schematic adsorption mode of arylidene barbiturate on mild steel in 1M HCl

The Corrosion inhibition of mild steel in hydrochloric acid solution by Arylidene barbiturates can be explained on the basis of adsorption as depicted in Figure 10. The inhibitor molecules interact with metal surface by:

(a) Protonated inhibitor molecules with already adsorbed chloride ions (Physical adsorption)

(b) Interaction of lone pair of electron of N atoms or π electrons of aromatic ring with vacant d-orbitals of the surface Fe atoms (Chemical adsorption).

(c) Interaction of d-electrons of Fe with the high energy orbitals of inhibitor molecules (Reterodonation) [31].

4. CONCLUSION

1. All the four Arylidene barbiturates show good inhibition efficiency in the range of 89-97% at 350 mgL^{-1}. The increasing order of inhibition performance of these compounds are in the order PPT> HPT> BPT> NPT.
2. All these compounds were found to affect both the anodic and cathodic processes and act as mixed-type inhibitors.
3. All the barbiturates inhibit corrosion by the adsorption mechanism.
4. The results obtained from weight loss and electrochemical methods are in good agreement.

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