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ORIGINAL PAPER

Investigation of triazole derived Schiff bases as corrosion inhibitors for mild steel in hydrochloric acid medium

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Abstract 3,5-Diamino-1,2,4-triazole Schiff base derivatives and their inhibition efficiency, based on the effect of changing functional groups, were reported to establish a relationship between inhibitor efficiency and molecular structure using weight loss method, electrochemical and Fourier transform infrared spectral techniques. It was found that the molecules containing more electron donating groups have higher inhibition efficiency than the corresponding compounds with low electron donating groups. The results indicate that the order of inhibition efficiency of the triazole and its Schiff bases in solution and the extent of their tendency to adsorb on mild steel surfaces are as follows: vanilidine 3,5-diamino-1,2,4-triazole > furfuraldine 3,5-diamino-1,2,4-triazole > anisalidine 3,5-diamino-1,2,4-triazole > 3,5-diamino-1,2,4-triazole.

Keywords Triazole · Schiff base · Mild steel · Corrosion · Potentiodynamic polarization

1 Introduction

Mild steel is widely used in many applications; this intensified the research in corrosion resistance in various aggressive environments [1–4]. In order to mitigate electrochemical corrosion, the primary strategy adopted is to isolate the metal from corrosive agents. Amongst the various methods available, the use of inhibitors is one of the most practical methods for protection against corrosion especially in acidic media [5–10]. Heterocyclic organic compounds containing nitrogen, sulphur or oxygen atoms are often used to protect metals from corrosion. For the past few years, azoles have been intensively investigated as effective corrosion inhibitors [11–17] for various environments.

Hydrochloric acid (HCl) is widely used in the acid pickling of steel and ferrous alloys, acid cleaning, acid rescaling, oil well cleaning, petrochemical industry and various electrochemical systems. Hence, much attention is needed on corrosion inhibitors to reduce the corrosion rates of metallic materials.

Triazoles and some Schiff bases have been reported earlier as corrosion inhibitors for steel, and they reveal that the inhibition efficiencies of the Schiff bases are much greater than that of the corresponding amines and aldehydes. There are few reports about the successful use of some polydentate Schiff base compounds containing aminic nitrogens as corrosion inhibitors for mild steel in acidic media [18–22]. It is found that the inhibition efficiencies of Schiff bases depend on the type and the nature of the substituents present in the molecule. Li et al. [23] have studied a few Schiff's bases derived from aromatic aldehydes and amines as corrosion inhibitors for copper in hydrochloric acid medium and similar observations have also been reported for mild steel corrosion [24–27]. These observations have prompted us to investigate the inhibiting behaviour of some Schiff bases derived from 3,5-diamino-1,2,4-triazole (TA).

In the present work, a systematic study has been undertaken to understand the corrosion behaviour of mild steel and the inhibitive action of TA and its Schiff base derivatives in 1 M HCl environment. Electrochemical techniques such as potentiodynamic polarization and electrochemical

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impedance have been employed to study the corrosion rate and inhibition efficiency. It was found that the adsorbed inhibitor on the mild steel surface accounts for protecting the metal from the corrosive medium and modifying the electrode reactions in favour of corrosion protection of the metal.

2 Experimental

2.1 Synthesis of Schiff base derivatives of 3,5-diamino-1,2,4-triazole

In this paper, Schiff bases having structures as shown in Fig. 1 were synthesized [28, 29]. The inhibitors, anisalidine 3,5-diamino-1,2,4-triazole (ATA), furfuraldine 3,5-diamino-1,2,4-triazole (FTA) and vanilidine 3,5-diamino-1,2,4-triazole (VTA) were prepared by condensation of TA with anisaldehyde, furfuraldehyde and vanillinaldehyde, respectively. All the chemicals chosen for our study were of analytical grade and double distilled water was used throughout the experiment. Further, the inhibitor solutions were prepared using 1 M HCl.

2.2 Specimen preparation

The mild steel specimens with the composition C-0.13%, P-0.032%, Si-0.014%, S-0.025%, Mn-0.48% and balance Fe were used in this study. For weight loss measurements, the metal specimens were mechanically cut into 4.0 cm × 2.0 cm × 0.2 cm dimension and polished with SiC abrasive papers (from grits 120 to 1200), and were further used in this study. For the electrochemical studies, specimens of size 1.0 cm × 1.0 cm × 0.3 cm were cut, embedded in the epoxy resin, polished, rinsed with double distilled water and dried.

2.3 Weight loss measurements

Mild steel specimens in triplicate were immersed in 1 M HCl for 2 h at room temperature (28 ± 2 °C) for each inhibitor concentration. Then the specimens were removed, rinsed in double distilled water, acetone and the loss in weight was determined. From this the inhibition efficiency (IE%) was calculated using the formula,

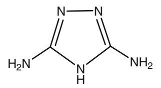
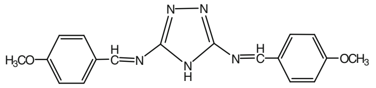
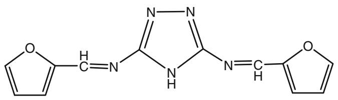
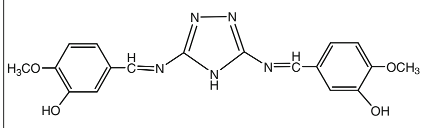
$$\mathrm{IE}(\%) = \frac{W_0 - W_i}{W_0} \times 100 \qquad (1)$$

where W_0 and W_i (in g) are the weight loss observed in the absence and presence of inhibitors.

2.4 Electrochemical studies

All the electrochemical measurements were performed using the Electrochemical Workstation (Model No: CHI 760, CH Instruments, USA) and a constant temperature of 28 ± 2 °C is maintained with 1 M HCl as an electrolyte. A platinum electrode and a saturated calomel electrode (SCE) were used as auxiliary and reference electrodes, respectively, while the working electrode comprised of mild steel specimens of 1 cm^2 exposed area. The tip of the reference electrode was positioned very close to the surface of the working electrode by the use of a fine Luggin capillary in

Fig. 1 Names and structures of the inhibitor compounds

Name of the inhibitor	**Abbreviation**	**Structure of the inhibitor**
3,5-diamino-1,2,4-triazole	TA	
Anisalidine 3,5-diamino-1,2,4-triazole	ATA	
Furfuraldine 3,5-diamino-1,2,4-triazole	FTA	
Vanilidine 3,5-diamino-1,2,4-triazole	VTA	

order to minimize the ohmic potential drop. The remaining uncompensated resistance was also reduced by the electrochemical workstation. Potentiodynamic polarization studies were carried out at a scan rate of 0.1 mV s^{-1} and at a potential range of −800 to −200 mV for various concentrations of inhibitors. The electrochemical impedance studies were carried out using the same set-up as that of potentiodynamic polarization studies and the applied ac perturbation signal was about 10 mV within the frequency range from 1 kHz to 1 Hz. All the electrochemical impedance measurements were carried out at open circuit potential.

The percentage of inhibition efficiency is calculated from the values of the corrosion current density (i_{corr}) with the aid of following formula:

$$\mathrm{IE}(\%) = \frac{(i_{\mathrm{corr}} - i_{\mathrm{corr'}})}{i_{\mathrm{corr}}} \times 100 \tag{2}$$

where i_{corr} and $i_{\mathrm{corr'}}$ are the values of corrosion current density of uninhibited and inhibited specimen, respectively.

2.5 Surface examination

The mild steel specimens were immersed in 1 M HCl containing the inhibitor VTA (0.30 mM) for 2 h after which they were taken out, washed with distilled water to remove the residual VTA, and dried then scraped the film using non-metallic scrapper. The Fourier transform infrared (FT-IR) spectra of the scraped films were recorded using a Nicolet 380 FT-IR spectrophotometer.

3 Results and discussion

3.1 Weight loss measurements

The corrosion of mild steel in 1 M HCl medium containing various concentrations of TA and its Schiff bases ATA, FTA and VTA (0.10, 0.15, 0.20, 0.25 and 0.30 mM) were studied by weight loss measurements. Figure 2 summarizes the inhibition efficiencies of the studied inhibitors at different concentrations. It is obvious from the figures that all these compounds inhibit the corrosion of mild steel in 1 M HCl solution at all concentrations used in this study and the inhibition efficiency was seen to increase with increasing additive concentration up to the optimum level after which there is no significant change in the inhibition efficiency. The studied inhibitors attain maximum IE(%) at 0.30 mM concentration. Thus, we deduce that these are good inhibitors for the mild steel corrosion and IE(%) are found to be in the following order: VTA > FTA > ATA > TA at their optimum concentration. The difference in their inhibitive action can be explained on the basis of the type of functional group present in the benzene ring which contributes to the adsorption strength through the donor acceptor bond between the non-bonding electron pairs and the vacant orbitals of the metal surface. In order to have better understanding of the inhibition mechanism of Schiff bases, a detailed study on corrosion inhibition of these compounds was carried out using potentiodynamic polarization and electrochemical impedance spectroscopy.

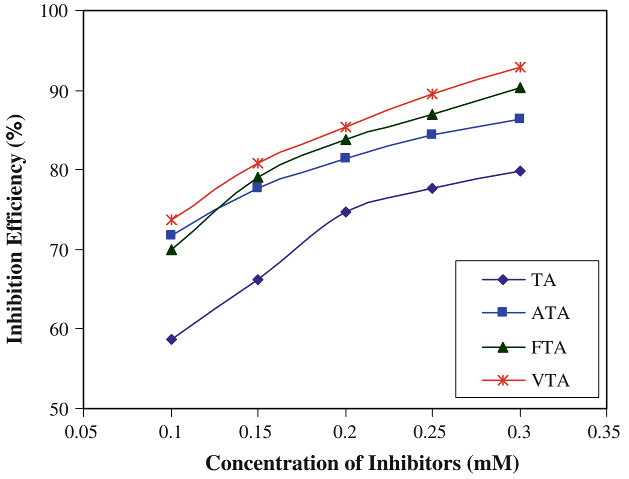


Fig. 2 Inhibition efficiency of TA and its Schiff bases ATA, FTA and VTA at various concentrations by weight loss measurement in 1 M HCl

3.2 Potentiodynamic polarization studies

Potentiodynamic polarization results obtained for the inhibitory effect of TA and its Schiff bases ATA, FTA and VTA on mild steel corrosion in 1 M HCl are shown in Figs. 3, 4, 5, 6 and 7. The associated corrosion parameters

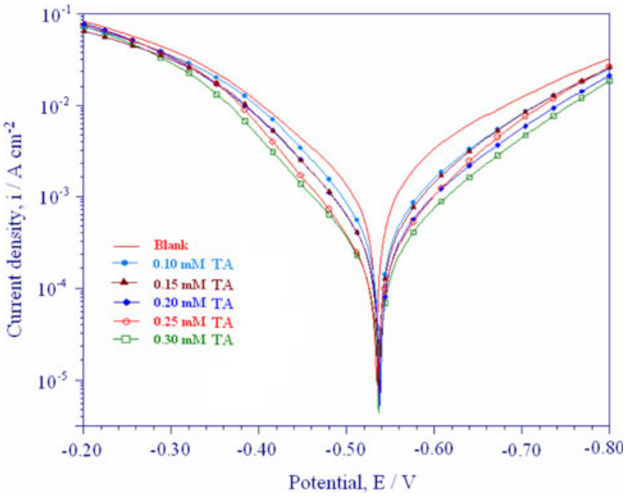


Fig. 3 Potentiodynamic polarization curves of mild steel in 1 M HCl in absence and presence of various concentrations of TA

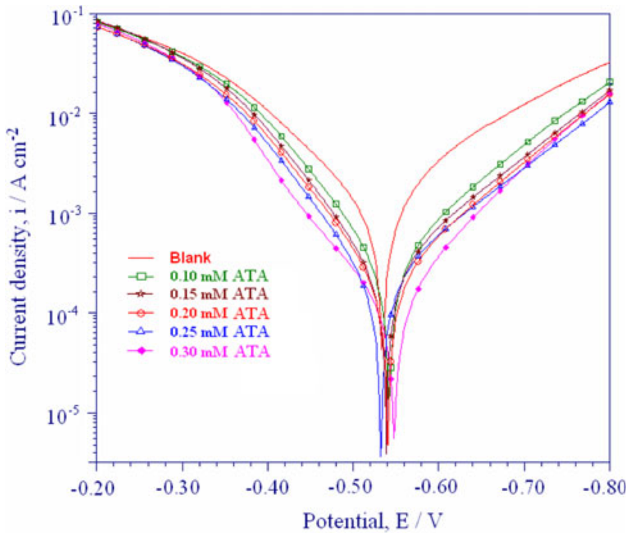


Fig. 4 Potentiodynamic polarization curves of mild steel in 1 M HCl in absence and presence of various concentrations of ATA

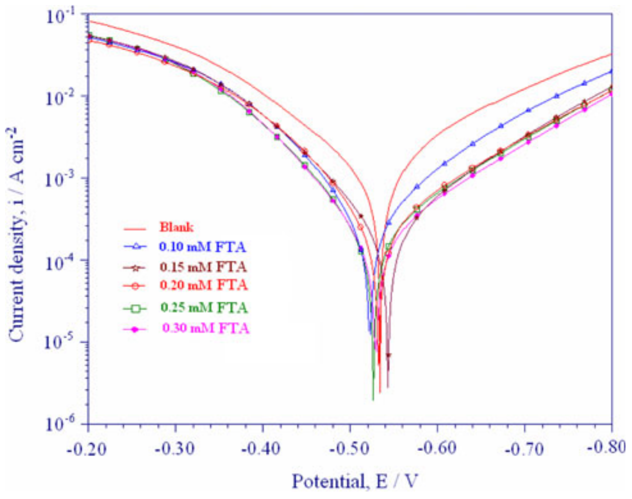


Fig. 5 Potentiodynamic polarization curves of mild steel in 1 M HCl in absence and presence of various concentrations of FTA

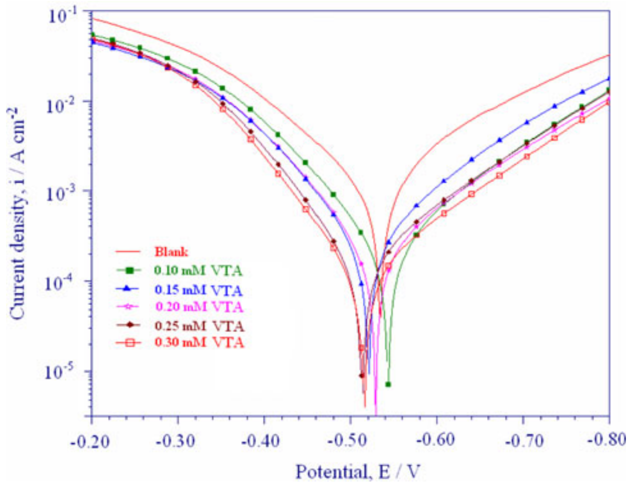


Fig. 6 Potentiodynamic polarization curves of mild steel in 1 M HCl in absence and presence of various concentrations of VTA

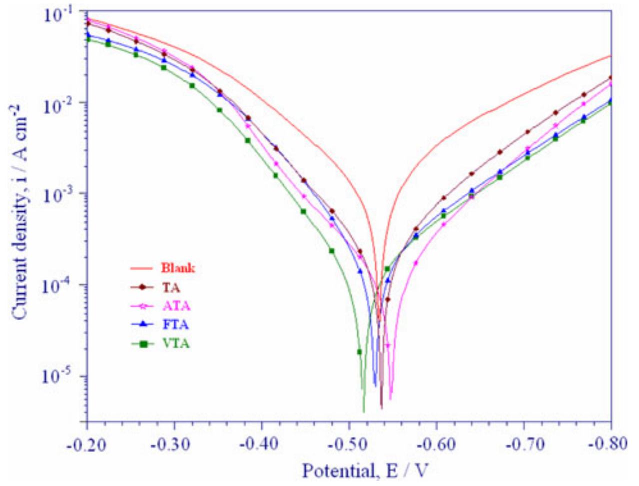


Fig. 7 Potentiodynamic polarization curves of mild steel in 1 M HCl in absence and presence of optimum concentration of TA, ATA, FTA and VTA

such as corrosion potential (E_{corr}), cathodic and anodic Tafel slopes (b_{c}, b_a) and corrosion current density (i_{corr}) are listed in Table 1. As shown in Fig. 3, the i_{corr} values obtained for the solution containing TA decreases with the increase in TA concentration up to 0.30 mM, after which no significant change in the i_{corr} values is noted. As it can be seen, both cathodic and anodic reactions of mild steel electrode were inhibited with the increase in TA concentration in 1 M HCl acid. The addition of TA decreases the i_{corr} values significantly for all the studied concentrations due to the increase in blocked fraction of the electrode surface by adsorption. It is also evident from this study that the inhibition of the mild steel corrosion is under both cathodic and anodic control and therefore TA can be classified as a mixed-type inhibitor.

Potentiodynamic polarization curves of mild steel in 1 M HCl in the absence and presence of various concentrations of ATA, FTA and VTA are shown in Figs. 4, 5 and 6, and the corresponding Tafel parameters are summarized in Table 1. Figure 4 shows the influence of various concentrations of ATA on the polarization behaviour of the mild steel in 1 M HCl. It should be noted that with the addition of the ATA both the anodic and cathodic current densities decrease and hence it also acts as a mixed-type inhibitors. In fact the other two inhibitors (FTA and VTA) were also found to act as a mixed type inhibitors since the i_{corr} values for mild steel in their presence decreased up to 0.30 mM and after which there is no significant change (Figs. 5, 6). The potentiodynamic polarization curves in the absence and presence of TA, ATA, FTA and VTA at their

Table 1 Potentiodynamic polarization parameters of mild steel in 1 M HCl in absence and presence of various concentrations of TA, ATA, FTA and VTA

Inhibitor	Concentration of inhibitor (M)	b_a (V dec^{-1})	b_c (V dec^{-1})	E_{corr} (V)	i_{corr} (mA)	Rate (mpy)	IE(%)
Blank	–	0.14	0.15	−0.5248	125.86	575.3	–
TA	0.10	0.10	0.12	−0.5370	51.86	237.0	58.79
	0.15	0.11	0.12	−0.5360	42.65	195.0	66.12
	0.20	0.11	0.12	−0.5539	34.62	158.2	72.49
	0.25	0.10	0.11	−0.5349	30.06	137.4	76.12
	0.30	0.11	0.12	−0.5376	27.64	126.4	78.04
ATA	0.10	0.09	0.12	−0.5218	34.73	158.5	72.58
	0.15	0.10	0.15	−0.5321	28.18	128.8	77.60
	0.20	0.10	0.13	−0.5432	23.33	106.7	81.45
	0.25	0.09	0.14	−0.5259	19.61	89.7	84.41
	0.30	0.09	0.14	−0.5290	17.25	78.9	86.29
FTA	0.10	0.10	0.17	−0.5322	39.02	178.4	68.99
	0.15	0.09	0.12	−0.5199	25.71	117.5	79.57
	0.20	0.09	0.14	−0.5288	21.57	98.6	82.86
	0.25	0.09	0.13	−0.5134	19.96	91.2	84.14
	0.30	0.08	0.14	−0.5149	12.09	55.3	90.39
VTA	0.10	0.10	0.13	−0.5414	31.62	144.4	74.87
	0.15	0.10	0.13	−0.5380	25.12	114.8	80.03
	0.20	0.10	0.13	−0.5410	20.36	93.0	83.82
	0.25	0.10	0.14	−0.5321	16.42	75.1	86.95
	0.30	0.10	0.09	−0.5474	9.051	41.4	92.81

optimum concentrations are shown in Fig. 7. From the potentiodynamic polarization studies, VTA shows the maximum inhibition efficiency (92.81%) among all the other studied compounds due to the maximum adsorption of VTA triggered by the high electron density of the nitrogen atom in the molecule. The excellent behaviour of VTA is also previously evidenced from the weight loss measurements. Thus, the inhibition efficiency follows the order VTA > FTA > ATA > TA.

The inhibition efficiency depends on many factors including adsorption centres, mode of interaction, molecular size and structure [30–32]. The effect of molecular size, structure and functional group can be easily viewed from the difference in the structure of VTA with respect to FTA and ATA.

3.3 Electrochemical impedance studies (EIS)

The corrosion of mild steel in 1 M HCl solution in the absence and presence of TA, ATA, FTA and VTA were investigated by EIS at the open circuit potential condition. Nyquist plots for mild steel obtained at the interface in the absence and presence of inhibitors at optimum concentration is given in Fig. 8. The Nyquist diagram obtained with 1 M HCl shows only one capacitive loop and the diameter of the semicircle increases on increasing the inhibitor concentration suggesting that the formed inhibitive film was strengthened by the addition of inhibitors. All the obtained plots show only one semicircle and they were fitted using one time constant equivalent model (Randle's model) with capacitance (*C*) and charge transfer resistance

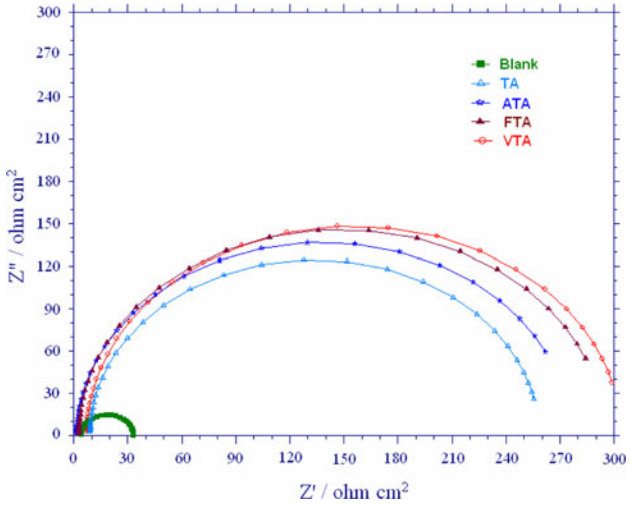


Fig. 8 Nyquist plots of mild steel in 1 M HCl in absence and presence of optimum concentration of TA, ATA, FTA and VTA

(R_{ct}). The corresponding Bode plots are shown in Fig. 9 and all the main parameters deduced from the impedance technique are given in Table 2.

The lower capacitance (C_{dl}) value for 1 M HCl medium indicates the inhomogeneity of surface of the metal roughened due to corrosion. The C_{dl} values decreases on increasing the inhibitor concentration and reaches very low value for the optimum concentrations of all the studied systems indicating that the reduction of charges accumulated in the double layer due to formation of adsorbed inhibitor layer [33]. The charge transfer resistance of double layer increases on increasing the concentration of the inhibitors upto the optimum level indicating the decreased corrosion rate (i.e., increased corrosion inhibition). The inhibition efficiencies calculated according to the impedance results are in the order: VTA > FTA > ATA > TA and these results follow the same trend as the polarization results.

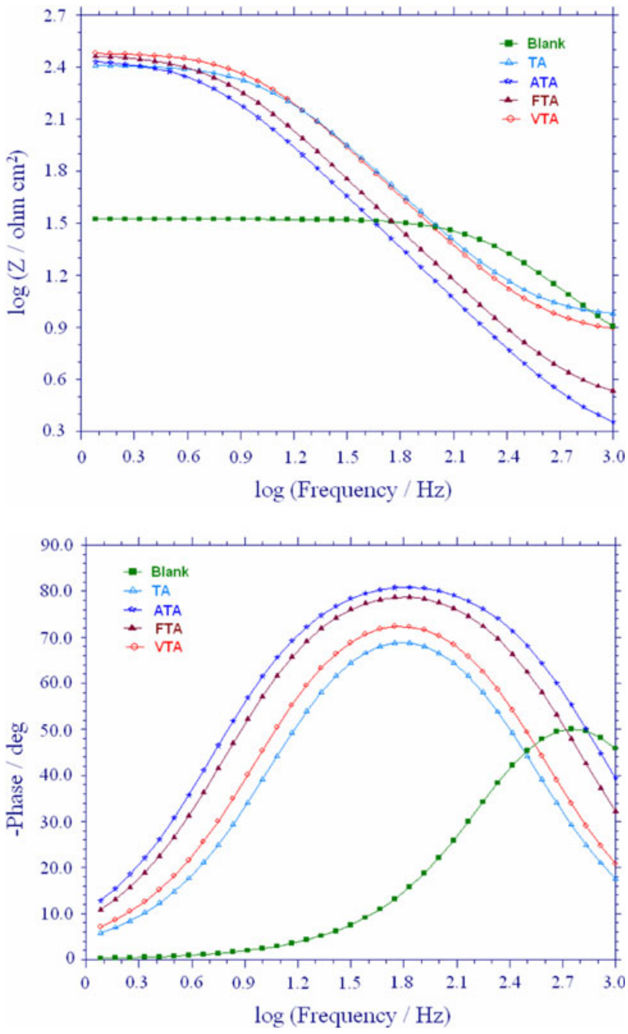


Fig. 9 Bode plots of mild steel in 1 M HCl in absence and presence of optimum concentration of TA, ATA, FTA and VTA

Table 2 Electrochemical impedance parameters obtained for mild steel in 1 M HCl in absence and presence of optimum concentration of TA, ATA, FTA and VTA

Inhibitor	R_S (Ω)	R_P (Ω)	C_{dl} (μF cm^{-2})
Blank	4.45	28.9	0.791
TA	2.36	245.2	0.131
ATA	1.75	272.3	0.113
FTA	1.52	287.4	0.097
VTA	1.28	299.2	0.082

The increase in inhibiting efficiencies with the increase in the concentrations of the studied Schiff bases show that the inhibitory actions are may be due to the adsorption of the inhibitors on steel surface. It is generally assumed that the adsorption of inhibitor at the metal/solution interface is the mechanism of inhibition through electrostatic attraction between the charged molecules and charged metal. Among the compounds investigated in the present study, VTA has been found to give an excellent inhibition due to the presence of the electron donating groups (such as OH and OCH_3) on the Schiff base structure, which increases the electron density on the nitrogen of the C=N group. This leads to the strong adsorption of VTA on the metal surface thereby resulting in high inhibition efficiency.

3.4 Adsorption isotherm

Basic information on the interaction between the inhibitor and the mild steel surface can be proved by the adsorption isotherm and in general, inhibitors can function either by physical (electrostatic) adsorption or chemisorption with the metal. To obtain more information about the interaction between the Schiff base molecules and the metal surface, different adsorption isotherms were tested. The fractional surface coverage θ at different concentrations of TA, ATA, FTA and VTA in 1 M HCl solution were determined from the potentiodynamic polarization measurements data [34] using the formula

$$\theta = \frac{i_{corr} - i_{corr(inh)}}{i_{corr}} \tag{3}$$

where i_{corr} and $i_{corr(inh)}$ are the values of corrosion current density of uninhibited and inhibited specimens, respectively.

$$Kc = \frac{\theta}{1-\theta} \tag{4}$$

where c is the concentration of the inhibitor, θ is the fractional surface coverage. The Langmuir isotherm, Eq. 4, which is based on the assumption that all adsorption sites are equivalent and that molecular binding occurs independently from the fact whether the nearby sites are occupied or not, was verified for all the studied Schiff bases. The adsorption equilibrium constant K is related to the free energy of adsorption ΔG_{ads} as

$$K = \frac{1}{C_{\mathrm{solvent}}} \exp\left(\frac{-\Delta G_{\mathrm{ads}}}{RT}\right) \tag{5}$$

where C_{solvent} represents the molar concentration of the solvent, which in the case of water is 55.5 mol dm^{-3}, R is the gas constant and T is the thermodynamic temperature in K. The Langmuir isotherm, Eq. 4, can be rearranged to obtain the following expression:

$$\frac{c}{\theta} = \frac{1}{K} + c \tag{6}$$

so that a linear relationship can be obtained on plotting c/θ as a function of c, with a slope of unity (Fig. 10). The thermodynamic parameters K and ΔG_{ads} for the adsorption of the studied inhibitors on mild steel is obtained by Langmuir's adsorption isotherm and are given in Table 3. The negative values of $\Delta G^0_{\mathrm{ads}}$ for the addition of inhibitors indicate that the process of adsorption of studied inhibitors are spontaneous in nature [35]. The free energy of adsorption of TA (ΔG_{ads}), in 1 M HCl was found to be −7.25 kJ mol^{-1} while for the Schiff bases (ATA, FTA and VTA) it was found to be −7.86, −8.31 and −8.65 kJ mol^{-1}, respectively.

It is well known that values of ΔG_{ads} in the order of −20 kJ mol^{-1} or lower indicate a physisorption while those about −40 kJ mol^{-1} or higher involve charge sharing or transfer from the inhibitor molecules to the metal surface to form a co-ordinate type of bond [36]. The calculated adsorption values for the triazole and its Schiff bases show that the adsorption is of physical in nature, and there is no chemical interaction between the inhibitor molecule and the metal surface. This indicates that the adsorption of TA, ATA, FTA and VTA takes place through electrostatic interaction between the inhibitor molecule and the metal surface.

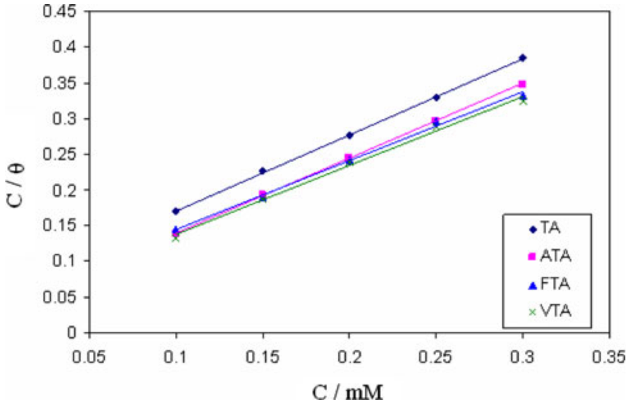


Fig. 10 Langmuir adsorption isotherm of mild steel in 1 M HCl containing TA, ATA, FTA and VTA at 301 K

Table 3 Thermodynamic parameters for the adsorption of TA, ATA, FTA and VTA in 1 M HCl on the mild steel at 301 K

Inhibitor	K (10^4 M^{-1})	ΔG_{ads} (kJ mol^{-1})
TA	0.49	−7.25
ATA	0.38	−7.86
FTA	0.30	−8.31
VTA	0.24	−8.65

3.5 FT-IR studies

A transmission vibrational spectrum of VTA is depicted in Fig. 11a. A peak at 1664 cm^{-1} indicates the presence of the C=N bond of the formed Schiff base and the appearance of a very weak band in the region of 3560 cm^{-1} corresponds to the O–H stretching vibration of the hydroxyl group. The presence of C–N and O–C stretching frequencies are clearly manifest in the region from 1200 to 1020 cm^{-1} and 1516 cm^{-1}, respectively. Further, peaks at 1531–1450 cm^{-1} can be attributed to the (N–N) group present in the ring and the C=C ring stretching peak appears at 1415–1360 cm^{-1}. The peaks for C–N stretching modes can be assigned in the region around 1160 cm^{-1}.

The transmission FT-IR spectra obtained for the mild steel specimens immersed in 1 M HCl containing the inhibitor VTA is presented in Fig. 11b. This shows the characteristic bands for the adsorbed VTA on the metal surface. A weak band in the range from 3000 to 3500 cm^{-1}, can be assigned to the presence of O–H. The presence of N–N, C=C, C–N and O–C is indicated by their stretching modes at 1690, 1500–1450, 1390 and 1120 cm^{-1}, respectively. Moreover, there are no additional peaks identified for the formation of Fe–inhibitor complex and this reveals that there is only physical adsorption occurred on the surface of the metal. This is already confirmed from the Langmuir adsorption isotherm studies.

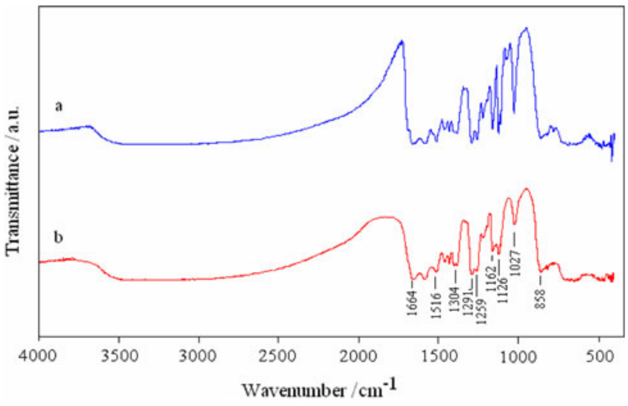


Fig. 11 FT-IR spectra of (*a*) VTA (*b*) mild steel immersed in 1 M HCl containing optimum concentration of VTA

4 Conclusions

The present study leads to the following conclusions in controlling the corrosion of mild steel by triazole derived Schiff bases in 1 M HCl environment;

1. TA and its Schiff bases (ATA, FTA and VTA) have been found to be good candidates of corrosion inhibitors for mild steel in 1 M HCl medium.
2. TA, ATA, FTA and VTA inhibit both anodic and cathodic reactions by adsorption on the mild steel surface and hence behave like mixed type inhibitors.
3. The inhibitor molecules adsorb on the mild steel surface and tend to retard the rate of corrosion by reducing the number of available surface sites for corrosion.
4. VTA exhibits the highest inhibition efficiency (92.81%) at 0.30 mM concentration for the mild steel corrosion in 1 M HCl due to the enriched electron density on C=N.
5. The results obtained from weight loss measurements and the electrochemical studies are complimentary to one another and have shown that VTA has the maximum inhibition efficiency than all the other studied systems.
6. The adsorption of all compounds investigated follows the Langmuir isotherm and the FT-IR results also reveals the adsorption of inhibitor molecule on the metal surface and blocking the active sites.

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