

5-(4-chlorophenyl)-1H-tetrazole as an effective corrosion inhibitor for mild steel used in oil and natural gas industries

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Abstract: The effect of 5-(4-chlorophenyl)-1H-tetrazole (Cl-PTZ) on corrosion inhibition of mild steel in 1M HCl was investigated by weight loss measurement, open circuit potential(OCP), potentiodynamic polarization and surface measurements. Results showed that the inhibition efficiency increased with increase in concentration of inhibitor and attained the maximum value at 500 ppm concentration. Polarization study revealed that 5-(4-chlorophenyl)-1H-tetrazole acted as mixed- type inhibitor. Among the several tested isotherms, adsorption of 5-(4-chlorophenyl)-1H-tetrazole on the mild steel surface obeyed the Langmuir adsorption isotherm. The adsorption of corrosion inhibitor on mild steel surface is well supported by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) study.

Keywords: corrosion, mild steel, tetrazole, ocp, SEM/EDX

I. INTRODUCTION

Corrosion is naturally occurring phenomena defined as the deterioration in quality of materials (usually metal) or its properties because of the reaction of material with its environment. Corrosion of material is inevitable due to the fundamental need of lowering of Gibbs free energy (Ossai CI.,2012;Elmsellem et al.,2015).Mild steel has been used extensively in the oil, gas and chemical industries because of its outstanding mechanical properties. The use of steel is also one of the effective strategies to maximize profit and reduce cost as compared to expensive corrosion resistant alloys. In many industrial applications related to oil and gas processing such as pipeline cleaning, pipeline/acid descaling and oil well acidizing, the use of mineral acids (usually hydrochloric acid) is still an effective method for improving productivity. This process

however endangers the life of steel structures as a result of acid driven corrosion. In order to prevent this undesirable reaction, corrosion inhibitors are often added to the acid solution during acidification process (Hassan AR., and Gbadeyan JA.,2015; Ferdows M., et al.2009).Organic compounds, containing functional electronegative groups and π -electron in triple or conjugated double bonds are usually good inhibitors (Cano E., et al 2004; Ghailane T., et al.2013). Heteroatoms, such as sulphur, phosphorus, nitrogen and oxygen, together with aromatic rings in their structure are the major adsorption centres (Goulart CM., et al.2013; Bentiss F., et al 2005). The planarity (g) and the lonely electron pairs in the heteroatoms are important features that determine the adsorption of these molecules on the metallic surface (Hegazy MA., et al.2013; Hu SQ., et al.2010). The use of corrosion inhibitors is one of the most effective measures for protecting metal surfaces against corrosion in acid environments (Issa RM., et al.2008; Khamis A., et al.2013).

Tetrazoles are one of the most stable nitrogen rich heterocyclic compounds among other heterocyclic systems. Due to their higher nitrogen content they show the exciting acidity, basicity, and complex formation constants. They have provided a platform for the rapid exchange of research in the area of organic, pharmaceutical, analytical,Material science and medicinal chemistry.Literature study revealed that the tetrazole ring, particularly, the 5-substituted-1H-tetrazole, that is, commonly referred as tetrazolic acid, has been widely employed in corrosion science as a corrosion inhibitors for protection of metals.

The present study was undertaken to investigate the inhibition of corrosion of mild steel in 1M hydrochloric acid by 5-(4-

chlorophenyl) tetrazole. The study was conducted by using gravimetric analysis, electrochemical technique such as Open circuit potential, potentiodynamic polarization and surface analytical techniques such as Scanning electron microscope (SEM) and Energy Dispersive X-ray spectroscopy (EDX) studies.

II. EXPERIMENTAL PROCEDURE

A. Material and sample preparation

The weight loss and electrochemical experiments were performed on mild steel specimen having chemical composition, C-0.16%, Si-0.10%, Mn-0.40%, P-0.013%, S-0.02% and remaining iron. The exposed dimensions were 3cm x 1cm x 0.1cm and 1cm² for the weight loss and electrochemical experiment, respectively. Before exposure to test solution, exposed areas were cleaned with different grades of emery paper, 1/0, 2/0, 3/0, and 4/0, washed with triple distilled water, degreased with absolute alcohol and finally dried and stored in desiccator. Specimens were weighed on an electronic balance (CONTECH, Model No. CBS-50, least count 0.001g). The aggressive solution was made of AR grade hydrochloric acid obtained from Merck chemicals. The organic inhibitor was received from sigma-Aldrich and used without further purification. The chemical structure of inhibitor is shown in figure 1.

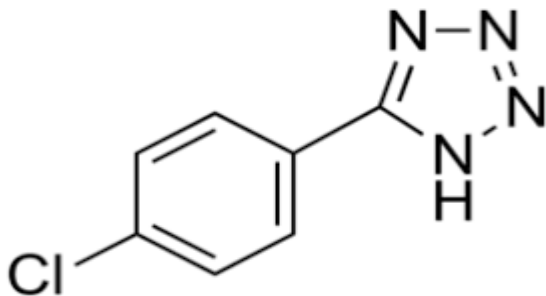


Fig.1. Structure of 5-(4-chlorophenyl)-1H-tetrazole (Cl-PTZ)

B. Weight loss measurement

Due to reliability and simplicity of weight loss method, it is preferably the starting method for corrosion testing. After initial weighing, the specimens were immersed in beakers which contained 100ml of 1M HCl in the absence and presence of different concentration of inhibitor. After 24 hrs, the specimens were taken out, washed, dried and weighed accurately. Mean corrosion rate (CR) in mg cm² h⁻¹ with respect to acid and inhibitor was calculated. The experiment was performed in triplicate to ensure reproducibility, and mean value was reported. The corrosion rate (CR) was calculated from following equation:

$$CR = \Delta W / At \quad (1)$$

Where CR is corrosion rate (mg cm²h⁻¹), ΔW is the weight loss (mg), A is surface area and t is immersion time in h. The inhibition efficiency (E%) of inhibitor on corrosion of mild steel was calculated as follows,

$$E \% = \frac{W_0 - W}{W_0} \times 100 \quad (2)$$

Where, W₀ and W are corrosion rate of mild steel in the absence and presence of inhibitor, respectively.

C. Electrochemical measurement

Polarization studies were carried out using electrochemical measurement system, DC 105, containing software of DC corrosion technique from M/S Gamry instruments, 734 Louis Drive, Warminster, PA, USA. For polarization studies, mild steel specimens having surface area of 1cm² were exposed to acid solution. Mild steel as working electrode, saturated calomel (SCE) as reference electrode and graphite as auxiliary electrode were used for all electrochemical measurements. Before each measurement the specimen were allowed to corrode freely, and their OCPs were measured as a function of time to obtain a steady-state potential. The anodic and cathodic Tafel curves were obtained by changing the electrode potential automatically from -0.5 to 0.5 V. at the scan rate of 5mv/s. polarization study was done with and without inhibitor in 1M HCl.

D. Surface measurement

The surface film formed on the metal specimen was evaluated by SEM-EDAX analysis. This was carried out by SEM/EDAX model PHENOM PROX from the Netherlands. The spectra were recorded for specimens exposed to 1M HCl for a period of 24 hours in the absence and presence of 500 ppm inhibitor. The energy of acceleration beam employed was 20 kv.

III. RESULTS AND DISCUSSION

A. Weight loss Measurement

Weight loss data of mild steel in 1M HCl in the absence and presence of various concentrations of inhibitor were obtained and are given in table I.

The result showed that decrease in weight loss and increase in inhibition efficiency was concentration dependant. The maximum inhibition efficiency was attained at 500 ppm, beyond this concentration there was no any remarkable change in the

inhibition efficiency for 24 h immersion period (Shukla J., and Pitre K.S., 2002; Ebenso EE. et al. 2010).

Table I: The weight loss parameter obtained for mild steel in 1M HCl containing different concentration of Cl-PTZ

Inhibitor	Conc. (ppm)	Weight Loss(mg)	CR $\text{mgcm}^{-2}\text{h}^{-1}$	Surface coverage (θ)	E(%)
Blank		168	23.33	-	-
	100	40	5.556	0.7619	76.19
Cl-PTZ	200	29	4.027	0.8273	82.73
	300	24	3.334	0.8571	85.71
	400	15	2.083	0.9107	91.07
	500	10	1.338	0.9404	94.04

B. Adsorption Isotherm

In corrosion field Adsorption isotherm provides basics information regarding interaction between the inhibitor molecules and metal surface (Dubey R.S., and Ambhore B.D., 2015); Behpour M., et al. 2014). To find suitable adsorption isotherm in the present study, several commonly used isotherms were tested, among which Langmuir adsorption isotherms were found to fit well with experimental data. Assumption of Langmuir relates the concentration of the adsorbate in the bulk of the electrolyte (C) to the degree of surface coverage (θ) according to equation:

$$C/\theta = 1/K + C \quad (3)$$

where K is equilibrium constant of adsorption. The graph of C/θ vs C obtained straight line with R^2 value obtained varied close to unity confirming the validity of this approach as shown in figure 2.

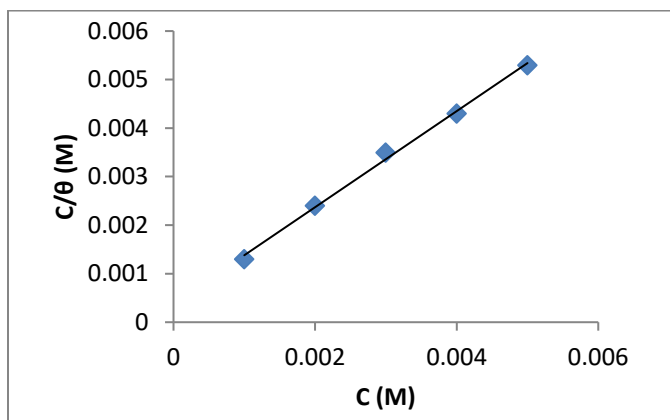


Fig. 2. Langmuir adsorption isotherm plot for the adsorption of Cl-PTZ on the mild steel surface in 1M HCl solution

The slope of the straight line was almost close to unity, suggesting that adsorbed molecule formed monolayer on mild steel surface (Varma C., et al. 2016; Chaitra TK., et al. 2016).

Free energy of the adsorption (ΔG_{ads}^0) can be calculated by using the following equation:

$$\Delta G_{\text{ads}}^0 = -RT \ln(55.5 k_{\text{ads}}) \quad (4)$$

Where R is the universal gas constant, T is the absolute temperature in K, and the numerical value 55.5 represents the molar concentration of water in the acid solution.

In the present case calculated value of ΔG_{ads} for the 5-(4-chlorophenyl)-1H-tetrazole was found to be -31.03 KJ/mol. This indicated that the adsorption of inhibitors on the mild steel surface involved both physisorption and chemisorption but physisorption was the predominant mode of adsorption. The negative value of ΔG_{ads}^0 indicated the spontaneous adsorption of tetrazoles on the surface of the metal (Chaitra TK., et al. 2015; Daoud D., et al. 2014).

C. Open Circuit Potential (OCP) Curves

The electrochemical behavior of mild steel in 1M HCl was studied by monitoring change in corrosion potential with time. The change in open circuit potential of mild steel in absence and presence of various concentration of inhibitor in 1M HCl is shown in figure 3.

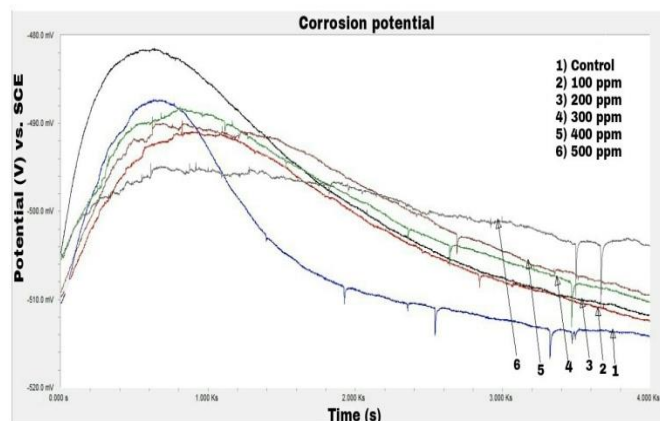


Fig.3. Open circuit potential of mild steel without and with different concentration of Cl-PTZ in 1M HCl

The change in OCP of mild steel in absence and presence of inhibitor was measured for period of one hour with sample period of one data per second. The potential attended steady state after exposure of approximately 30 minutes. The steady state potential is an equilibrium state at which I_{ox} is equal to I_{red} . It was observed that OCP of mild steel from moment of immersion in 1M HCl tends towards more negative value in

absence of inhibitor. This showed corrosiveness of medium which was due to breakdown of pre-immersion, air formed oxide film on the metal surface. In the presence of various concentrations of inhibitor the steady state potential of mild steel shifted more towards positive value. This was due to adsorption of inhibitor on metal surface resulting in passivation of metal [Singh AK., and Quraishi MA., 2010; Arab ST., and Al-Turkustani AM., 2006].

D. Potentiodynamic Polarisation Studies

In order to know the kinetics of anodic and cathodic reactions, polarization experiments were carried out potentiodynamically in unstirred 1 M HCl solution in the absence and presence of different concentration of **5-(4-chlorophenyl)-1H-tetrazole** and the obtained polarization curves are shown in Figure 4. Electrochemical parameters such as corrosion current (i_{corr}), corrosion potential (E_{corr}), and Tafel slope constant β_a and β_c calculated from Tafel plots are given in table II.

Table II: Polarization curves for corrosion of mild steel in 1 M HCl in the absence and presence of different concentration of Cl-PTZ

Conc. (ppm)	E_{corr} (mv)	I_{corr} ($\mu\text{A}/\text{cm}^2$)	β_a (v/dec)	β_c (v/dec)	η %
1M HCl	-470	548	$87.70 e^{-3}$	$164.4 e^{-3}$	--
5-(4-chlorophenyl)-1H-tetrazole					
100	-466	133.0	$68.20 e^{-3}$	$133.9 e^{-3}$	75.73
200	-462	71.00	$61.20 e^{-3}$	$129.1 e^{-3}$	87.04
300	-488	67.30	$67.40 e^{-3}$	$156.8 e^{-3}$	87.72
400	-491	41.70	$74.50 e^{-3}$	$171.1 e^{-3}$	92.39
500	-493	36.90	$66.80 e^{-3}$	$164.8 e^{-3}$	93.27

From the evaluated i_{corr} value, $\eta\%$ can be calculated using the relation

$$\eta\% = \frac{i_0 - i_{\text{corr}}}{i_0} \quad (5)$$

Where i_0 and i_{corr} are the corrosion current densities in the absence and presence of inhibitor. From the results in the table, it can be observed that the values of corrosion current density (I_{corr}) of mild steel in the inhibitor-containing solutions was lower than those for the inhibitor-free solution. As seen from

Figure 4, the anodic and cathodic Tafel slopes were almost unchanged, indicated that the inhibition action of the inhibitor was not through the interference on the reactions of metal dissolution and reduction of protons. This indicated that the 5-(4-chlorophenyl)-1H-tetrazole acted as adsorptive inhibitor, i.e., it reduced anodic dissolution and also retarded the hydrogen evolution reaction via blocking the active reaction sites on the metal surface or even can screen the covered part of the electrode and therefore protected it from the action of the corrosion medium. In this way, it was expected that the inhibition efficiency ($\eta\%$) is increased with concentration of inhibitor [Fouda AS., et al.2014; Farsak M., et al. 2015].

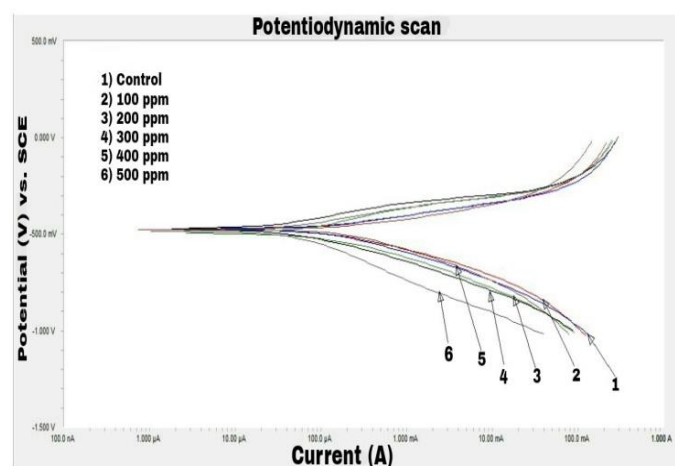


Fig. 4. Potentiodynamic polarization of mild steel without and with different concentration of Cl-PTZ in 1M HCl

According to the literature survey, it has been reported that if the displacement in E_{corr} in the presence of inhibitor is more than 85 mV with respect to E_{corr} of the blank, the inhibitor can be recognized as cathodic or anodic type. On the other hand, if the displacement in E_{corr} is less than 85 mV, the inhibitor can be classified as a mixed type. However, in the present study, the largest displacement exhibited by the inhibitors was less than 85 mV, from which it can be concluded that the inhibitor acted as mixed type [Kumar P., et al.2017; Ma H., et al.2003].

E. Scanning Electron Microscopic (SEM) Analysis

Tetrazole form protective film on surface of mild steel which is further supported by SEM micrographs of the mild steel surface obtained after immersion in 1M HCl for 24 hours in absence and presence of 500ppm concentration of tetrazole. Figure 5[A] represents the surface of mild steel without any addition. From figure 5[B] it can be seen that the specimen surface is rough and strongly damaged in the presence of acid

solution. Figure 5 [C] shows SEM micrograph of the mild steel surface immersed in 1M HCl containing 500 ppm of tetrazole. So we can conclude that the extent of damage to the mild steel surface is small and rate of corrosion is greatly reduced in presence of tetrazole. This was due to formation of protective surface film on mild steel, which acted as barrier and was responsible for inhibition of corrosion

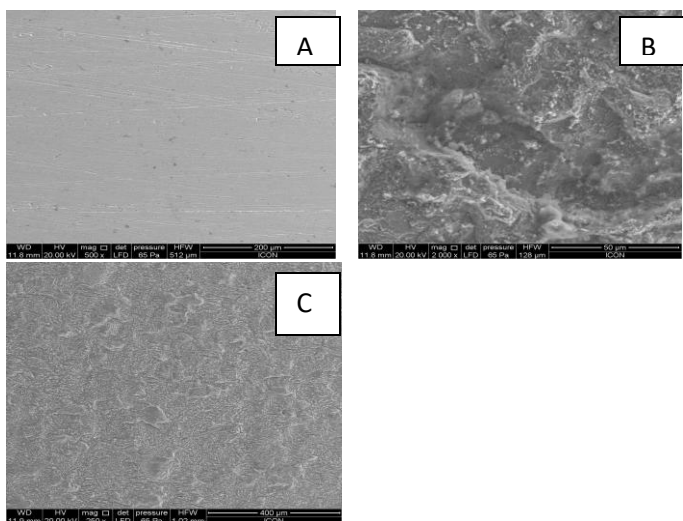
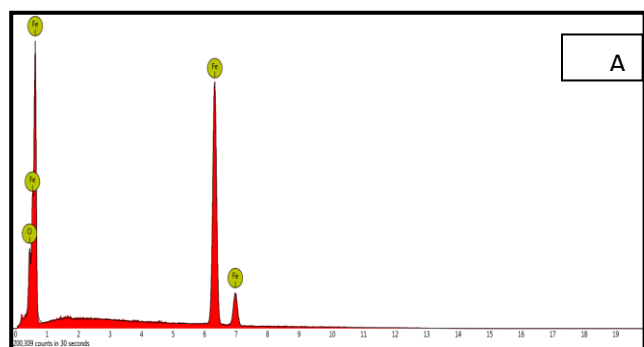


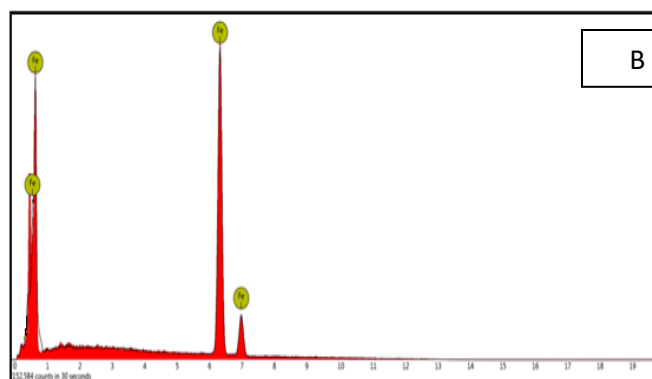
Fig. 5. SEM of mild steel with (A) Polished, (B) 1M HCl, (C) 500 ppm of Cl-PTZ

F. Energy-dispersive X-ray spectroscopy (EDX)

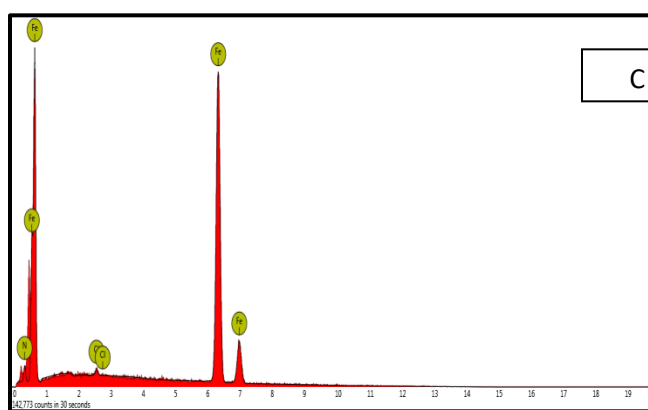
To support weight loss and electrochemical studies, EDX spectra were obtained from mild steel in the presence and absence of 500 ppm inhibitor.



A) Polished sample



B) Control (1M HCl)



C) 1M HCl+500 ppm Cl-PTZ

Fig. 6.EDX of mild steel with: (A)Polished, (B) 1M HCl, (C) 500 ppm of Cl-PTZ

Figure 6 [A] shows EDX spectra of polished mild steel surface with the characteristic peak for Fe and oxygen. From figure 6[B] it is clear that the EDX spectra in absence of tetrazole shows characteristic signal for Fe only as peak for oxygen disappear due to breaking of air formed oxide film in presence of 1 M HCl. However in presence of tetrazole the EDX spectra shown in figure 6 [C] indicates some additional peaks for nitrogen and chlorine confirming the presence of these elements on surface, which could be due to adsorption of studied inhibitor on mild steel surface.

IV. MECHANISM OF INHIBITION

The inhibition effect of tetrazole on corrosion of mild steel in 1 M HCl solution may be attributed to the adsorption of this compound to the metal solution interface. The extent of adsorption of the inhibitor depends on the nature of metal, chemical structure of inhibitor, nature of electrolyte and temperature (El-Maksoud, SA., 2008), Adsorption on the metal

surface was suggested to be via the active center present on inhibitor molecule and relies on their charge density (Hasanov R., et al.2007; .Moussa MN., et al.2007; Obot IB., et al.2009)

In case of tetrazole, the inhibition effect is due to interaction of π electron of phenyl and tetrazole ring as well as the presence of electron donor nitrogen through which it forms a bond with vacant d- orbital of the atom of the metal at the interface.

Generally, two modes of adsorption are considered on the metal surface in acid media. In first mode, the neutral molecules may be adsorbed on the surface of mild steel through the chemisorption mechanism, involving the displacement of water molecules from the mild steel surface and the sharing electrons between the hetero atoms and iron (Chakravarthy MP., and Mohana KN., 2014). The inhibitor molecules can also get adsorbed on the mild steel surface on the basis of donor-acceptor interactions between their π -electrons and vacant d-orbitals of surface iron. In second mode, since it is well known that it is difficult for the protonated molecules to approach the positively charged mild steel surface due to the electrostatic repulsion (Behpour M., et al.2008). Since Cl have a smaller degree of hydration, they could bring excess negative charges in the vicinity of the interface and favour more adsorption of the positively charged inhibitor molecules, the protonated inhibitor adsorb through electrostatic interactions between the positively charged molecules and the negatively charged metal surface. Thus, there is a synergism between adsorbed Cl ions and protonated inhibitors (Ahmad I., and Qurashi MA.,2009; Avci G.,2008).

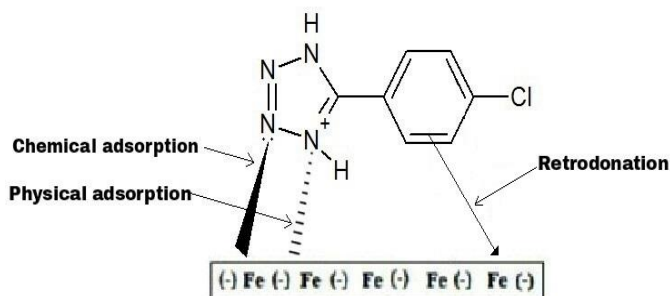


Fig.7.Pictorial representation of adsorption of Cl-PTZ on MS surface in 1M HCl

V. CONCLUSION

The results proved that 5-(4-chlorophenyl)-1H-tetrazole displayed good corrosion inhibition for mild steel in 1M HCl solution. The inhibitor acted as mixed type and shifted both cathodic and anodic curves in positive direction. The percentage inhibition efficiency increased with increase in

concentration of inhibitor. The data obtained from weight loss techniques fitted well in to the Langumir adsorption isotherm. The negative values of ΔG_{ads}^0 indicated spontaneous adsorption of the inhibitors on the mild steel surface. SEM-EDX analysis showed that there was improvement in surface morphology of inhibited mild steel as compared with uninhibited mild steel. so 5-(4-chlorophenyl)-1H-tetrazole found to be good inhibitor, as confirmed by all the chemical and electrochemical measurements.

CONFLICTS OF INTEREST

The authors declare no conflict of interest in the present research work.

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