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Adsorption and corrosion inhibitive properties of 5-(2-hydroxyphenyl)-1,3,4-oxadiazole-2-thiol on mild steel in acidic environment

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Abstract

The inhibition effect of 5-(2-hydroxyphenyl)-1,3,4-oxadiazole-2-thiol (5-HOT) on the corrosion of mild steel in 1M HCl, 2M HCl, 0.5M H_2SO_4 and 1M H_2SO_4 solution was investigated by electrochemical methods (potentiodynamic polarization curves, resistance polarization and electrochemical impedance spectroscopy). The obtained results show that the inhibitor under investigation acts as a very good inhibitor, and is more efficiency in 1M HCl than the other medium. The order is 1M HCl> 0.5M H_2SO_4 >1M H_2SO_4 > 2M HCl The adsorption of 5-HOT on mild steel surface obeys Langmuir adsorption isotherm for 1M HCl, 2M HCl and 0.5M H_2SO_4, and El Awady kenetic-thermodynamic isotherm for 1M H_2SO_4. Polarization curves reveal that 5-HOT acts as a mixed-type inhibitor in both acids. The adsorption mechanism is discussed.

Keywords: Acid medium, mild steel, corrosion, inhibition, oxadiazole-2-thiol derivative

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1. Introduction

For the removal of undesirable scale and rust in several industrial processes, the acid solutions are generally used. Hydrochloric and sulfuric acids are widely used in the pickling processes of metals and alloys. One of the most practical methods for protection against corrosion especially is use of inhibitors [1-2]. A number of organic compounds have been reported as effective corrosion inhibitors [3–8]. The more effective as corrosion inhibitors in acid media are the sulphur, oxygen and nitrogen containing compounds [9-13]. In view of the high performance of 1,3,4-oxadiazole compounds we have investigated the inhibitive effect of 5-(2-hydroxyphenyl)-1,3,4-oxadiazole-2-thiol (5-HOT) on corrosion of mild steel in HCl and H_2SO_4 electrochemical methods such as :polarization resistance, Tafel polarization and electrochemical impedance techniques.

2. Materials and methods

5-(2-hydroxyphenyl)-1,3,4-oxadiazole-2-thiol (5-HOT) was synthesized according as described in the literature [14-16]. Fig. 1 shows the molecular structure of 5-HOT.

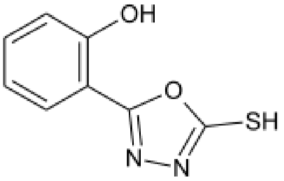

Fig. 1. Molecular 5-(2-hydroxyphenyl)-1,3,4-oxadiazole-2-thiol (5-HOT)

The electrochemical study was carried out using a potentiostat PGZ100 piloted by Voltamaster software. This potentiostat is connected to a cell with three electrode thermostats with double wall (Tacussel Standard CEC/TH). A saturated calomel electrode (SCE) is used as reference and platinum electrode is used as reference and auxiliary electrodes. Table 1 gives the composition of the material used as working electrode.

The surface area of working electrode exposed to the electrolyte is 0.04 cm^2. Prior to all measurements, the exposed area was mechanically abraded with different grades of emery papers.

Potentiodynamic polarization curves were plotted at a polarization scan rate of 0.5 mV/s. Before all the experiments, the potential of the metal was left at its abundance (free potential) for 30 min. The polarisation curves are obtained from cathodic to anodic branche (−800 mV to −200 mV) at 298 K. The solution test is there after de-aerated by bubbling nitrogen. Gas bubbling is maintained prior and through the experiments.

The polarization resistance measurements were performed by applying a controlled potential scan over a small range typically ±10 mV compared to E_{corr}. The resulting current is linearly plotted versus potential, the slope of this plot at E_{corr} being the polarization resistance (R_p).

The electrochemical impedance spectroscopy (EIS) measurements are carried out with the same electrochemical system. After the determination of steady-state current at a corrosion potential, sine wave voltage (10 mV) peak to peak, at frequencies between 100 kHz and 10 mHz are superimposed on the rest potential. Computer programs automatically controlled the measurements performed at rest potentials after 0.5 hour of exposure at 298 K. The impedance diagrams are given in the Nyquist representation. Experiments are repeated three times to ensure the reproducibility.

Values of the charge transfer resistance R_t were obtained from these plots by determining the difference in the values of impedance at low and high frequencies as suggested by Tsuru and Haruyama [17]. Values of the double–layer capacitance C_{dl} were calculated from the frequency at which the impedance imaginary component $-Z_i$ is maximum using the equation:

$$f(-Zi_{max}) = \frac{1}{2\pi C_{dl} R_t}$$

The corrosion current densities were determined by Tafel extrapolation of the cathodic curves to the E_{corr}. The inhibition efficiency was evaluated from the measured I_{corr} values using the relationship:

$$IE(\%) = I_{0corr} - \frac{I_{0corr}}{I_{0corr}} \times 100$$

where I_{icorr} and I_{0corr} are the corrosion current density in the presence and in the absence of inhibitor, respectively. The following equation gives the inhibition efficiency found out from the polarization resistance:

$$IE(\%) = R_{ip} - \frac{R_{0p}}{R_{ip}} \times 100$$

where R_{ip} and R_{0p} are the polarization resistance in the presence and in the absence of inhibitor, respectively. The inhibition efficiency of the inhibitor has been found out from the charge transfer resistance values using the following equation:

$$IE(\%) = R_{it} - \frac{R_{0t}}{R_{it}} \times 100$$

where R_{0t} and R_{it} are the charge transfer resistance in absence and in presence of inhibitor, respectively.

3. Results and discussion

3.1. Polarization measurements

Anodic and cathodic polarization curves in the absence and in the presence of 5-HOT at different concentrations after 30 min of immersion in 1M HCl, 2M HCl, 0.5M H_2SO_4 and 1M H_2SO_4 at 298 K are shown in Figs. 2-5.

From Figs. 2-5, in the cathodic part, we remark clearly that the cathodic current density decreases with increasing the concentration of the inhibitor; this phenomenon indicates that the 5-HOT is adsorbed on the metal surface and hence inhibition occurs. The value of b_c changed slightly with changing the inhibitor concentration in different medium, which indicates the presence the inhibitor molecules has no influence on the kinetics of hydrogen evolution. The presence of the molecules inhibitor moved slightly E_{corr} towards more negative potentials; it should be noted that this displacement in E_{corr} is < 85 mV. This implies that the inhibitor, 5-HOT, acts as a mixed-type inhibitor, affecting both anodic (dissolution of mild steel) and cathodic (release of hydrogen) reactions [18-20].

On the other hand, especially in 2M HCl, for anodic polarisation curves, it seems the presence of the inhibitor does not change the current versus potential for working electrode characteristics (the potential superior to -350 mV/SCE), this potential can be defined as desorption potential [20]. As it can be seen from these curves, the I_{corr} values decrease considerably in the presence of molecules inhibitor (5-HOT) and decreased with increasing inhibitor concentration (Table 2). We notice that the inhibition efficiency increased with inhibitor concentration. The latter reaches the values of 94.52%; 90.08; 89.82% and 79.67 at 10^{-3}M in 1M HCl, 0.5M H_2SO_4, 1M H_2SO_4, and 2M HCl, respectively.

Linear polarization technique was carried out with and without 5-HOT in different medium. The corresponding data are also given in Table 3. We can clearly see that the R_p values increases with increasing 5-HOT concentration. The inhibition percentage ($I.E_{Rp}$) calculated from R_p values are also presented in Table 3. We remark that $I.E_{Rp}$ increases with increasing concentration of inhibitor and attains 93.40%; 86.31%; 86.24% and 76.70% at optimum concentration (10^{-3}M) in 1M HCl, 0.5M H_2SO_4, 1M H_2SO_4, and 2M HCl respectively.

3.2. Electrochemical impedance spectroscopy

With the scope to understanding the kinetics and characteristics of the electrochemical process on the system: mild steel/acidic media and to get a better insight into the mechanism of corrosion and corrosion inhibition, electrochemical impedance spectroscopy (EIS) measurements were carried out. It should be noted that, EIS is a non-destructive technique that is used for the rapid characterization and study of corrosion inhibition behavior. For the system studied, Nyquist plots obtained in the absence and presence of 5-HOT at different concentration after 30 min of immersion and at 298 K are shown in Figs. 6-9.

We can clearly see that, all the plots display a single capacitive loop. The equivalent circuit used to fit the experimental data is Fig. 10. Impedance parameters derived from the Nyquist plots after fitting and percent inhibition efficiencies are listed in Table 4.

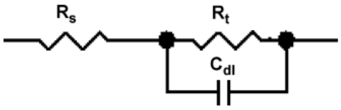

Fig. 10. The electrochemical equivalent circuit used to fit the impedance spectra.

It can be seen from table 4, that the presence of molecules inhibitors enhances the values of R_t and reduces the C_{dl} values. Several authors [21-23] attributed the decrease in C_{dl} as result from a decrease in local dielectric constant and/or an increase in the thickness of the electric double layer [24]. This ascertainment suggested that 5-HOT molecules function by adsorption at the metal/solution interface. Thus, the decrease in C_{dl} values and the increase in R_t values and consequently of inhibition efficiency may be due to the gradual replacement of water molecules by the adsorption of the 5-HOT molecules on the metal surface, decreasing the extent of dissolution reaction [24]. These impedance measurements were in good agreement with polarization tests.

3.3. Adsorption isotherms

The adsorption isotherm can provide important information on the interaction between the inhibitor molecules and the metal surface [25] and mechanism of electrochemical reaction [26-27]. In this study, the polarization curve measurements were used to calculate the surface coverage of different concentrations of 5-HOT in different acidic media. Several adsorption isotherms were examined, and it was found that the adsorption of 5-HOT in 1M HCl, 2M HCl and 0.5M H_2SO_4 at 298 K obeyed the Langmuir adsorption isotherm and in 1M H_2SO_4 El Awady kenetic-thermodynamic adsorption isotherm model was the most suitable model. It can be noted that, Langmuir [28] adsorption isotherm is given by:

$$\frac{c}{\theta} = \frac{1}{K_{ads}} + C$$

where C is the concentration of inhibitor, K_{ads} is the adsorption equilibrium constant and θ is the surface coverage. And El Awady kinetic-thermodynamic adsorption isotherm [29] is defined as bellow:

$$\ln\frac{\theta}{1-\theta} = \ln K' + y\ln C$$

where y is the numbers of inhibitor molecules occupying one active site. If the value of y is greater than one, it implies the formation of multilayer film on the metal surface, and if the values of y are less than one, it indicates that the studied inhibitor would occupy more than one active site. K' is a constant related to the equilibrium constant of adsorption process (K_{ads}) by following equation:

$$K_{ads} = K'^{(\frac{1}{y})}$$

From Fig. 11 it can be noted that the plots of C/Ө *vs* C (for 1M HCl, 2M HCl and 0.5M H_2SO_4) yielded straight lines with slope closed to one, along with linear association coefficient (R>0.98). The kinetic–thermodynamic isotherm is presented in figure 12.

In addition, the free adsorption energy (ΔG_{ads}) can be calculated according to the following equation:

$$\Delta G_{ads} = -RT\ln(55.5\ K_{ads})$$

where R is the universal gas constant, T is the absolute temperature. The thermodynamic parameters obtained by these adsorption isothrem are listed in table 4. From this table, we can see that the obtained values of 1/y is lower than one showing that a given multiple molecules inhibitor occupies one active site. This speculation was confirmed by the fact that the adsorption of 5-HOT did not obey Langmuir monolayer adsorption in 1M H_2SO_4. However, 5-HOT in 1M HCl, 2M HCl and 0.5M H_2SO_4 obeyed the Langmuir isotherm and formed a single layer on the metal surface, which can be explanation for better corrosion inhibition efficiency of MTI in these medium [1-2]. From the same table, it was found that the values of ΔG_{ads} were negative, which meant the spontaneity of the adsorption process and the stability of the adsorbed layer [4-5].

Generally, the electrostatic interaction between the charged molecules and the charged metal (physical adsorption) was evaluated by ΔG_{ads} upto -20 kJ/mol while those more negative than -40 kJ/mol is characteristic chemisorption [30-31]. In the present study, the value of ΔG_{ads} is between -34.49 and -28.40kJ/mol in 1M HCl, 2M HCl and 0.5M H_2SO_4; probably mean that the adsorption mechanism of the MTI on mild steel in different medium is mainly the chemisorptions and physical adsorption. Noticeably, it is generally accepted that physical adsorption is preceding stage of chemisorption of inhibitor molecules on metal surface. On the other hand, it is clearly that in 1M H_2SO_4 the adsorption is physical.

3.4. Explanation of adsorption and corrosion inhibition

We can clearly see that, the inhibitor studied is more efficient in HCl medium than in H_2SO_4 medium. This difference is due to the difference of the counter ions in the solution and their adsorption on the surface of mild steel (active sites). These latter leave fewer sites for organic molecules [32]. On the other hand, and according to several researchers, the Cl^- anion facilitates the adsorption of inhibitor molecules [33-34]. This explains the higher inhibitory efficacy of 5-HOT in 1M HCl medium compared to that obtained in 0.5M H_2SO_4 medium.

It should be noted that it is the adsorption of the inhibitory molecules at the metal/solution interface, forming a protective film which is transformed by a transition of the metal/solution interface from a state of active dissolution to the passive state. The reactive metal surface is protected from the aggressive environment by a rapid adsorption rate.

It is well known that inhibitory organic molecules establish their inhibitory action with the binding of inhibitory molecules to the metal/solution interface. Therefore, it is accepted that the adsorption process occurs by the replacement of solvent molecules existing on the surface by ions and molecules accumulated in the vicinity of the metal/solution interface. Moreover, the ions can accumulate at the metal/solution interface beyond those necessary to balance the charge on the metal at the operating potential [35-36].

Aromatic compounds like the case of our inhibitor (5-HOT) undergo particularly strong adsorption on many electrode surfaces. The bonding can occur between metal surface atoms and the aromatic ring of the adsorbate molecules or ligands substituent groups (mercapto and hydroxyl groups). The exact nature of the interactions between a metal surface and an aromatic molecule depends on the relative coordinating strength towards the given metal of the particular groups present [10].

In general, owing to the complex nature of adsorption and inhibition of a given inhibitor, it is impossible for single adsorption mode between inhibitor and metal surface. The adsorption of the 5-HOT can be attributed to the presence of hetero atoms (N, O and S) and aromatic rings. Therefore, the possible reaction centers are unshared electron pair of heteroatoms and π-electrons of aromatic ring.

Two modes of adsorption are generally considered on the surface of metal. In one mode, the neutral 5-HOT may be adsorbed on the surface of mild steel through the strong adsorption (chemisorption mechanism), involving the displacement of water molecules from the mild steel surface. The inhibitor molecules can also adsorb on the mild steel surface on the basis of donor–acceptor interactions between-electrons of the aromatic ring and vacant d-orbitals of surface steel atoms. In second mode, since it is well known that the steel surface bears positive charge in acid solution [4], so it is difficult for the protonated 5-HOT to approach the positively charged mild steel surface (H_3O^+/metal interface) due to the electrostatic repulsion. Since chloride ions have a smaller degree of hydration, thus they could bring excess negative charges in the vicinity of the interface and favour more adsorption of the positively charged inhibitor molecules, the protonated 5-HOT 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 5-HOT molecules. The presences of electron donating group (SH and OH) in the aromatic rings increase the electron density on nitrogen and oxygen of oxadiazole group, resulting high inhibition efficiency.

Table 1. Composition of working electrode

Elements	C	Si	Mn	Cu	S	Fe
%	0.18	0.16	0.44	0.20	0.03	Balance

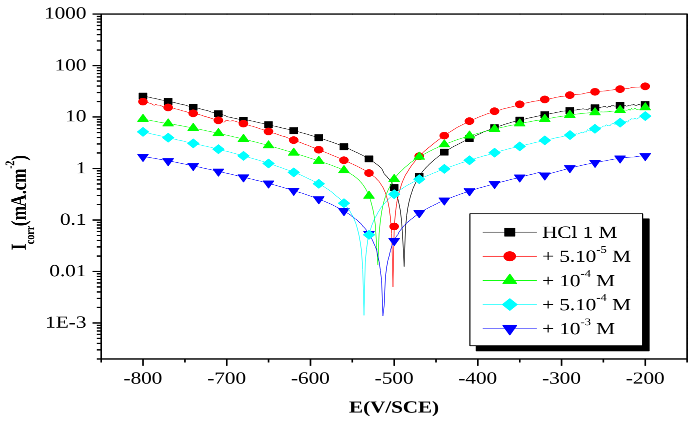

Fig. 2. Polarization curves for the system: mild steel/1M HCl containing 5-HOT

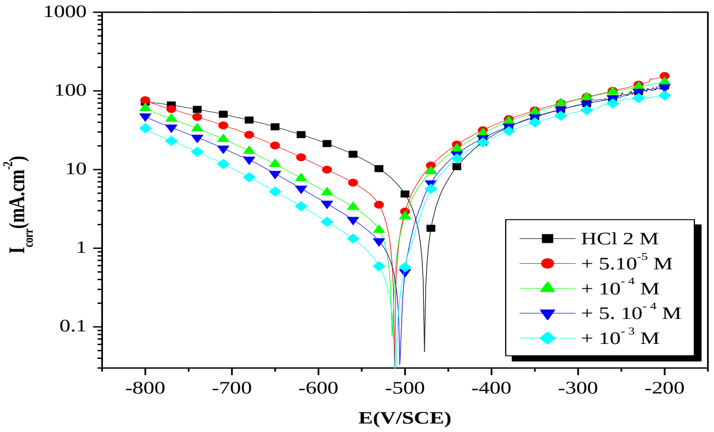

Fig. 3. Polarization curves for the system: mild steel/2M HCl containing 5-HOT

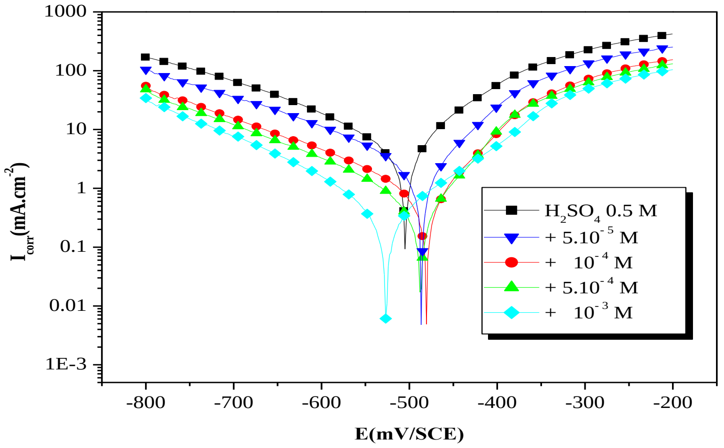

Fig. 4. Polarization curves for the system: mild steel/0.5M H_2SO_4 containing 5-HOT

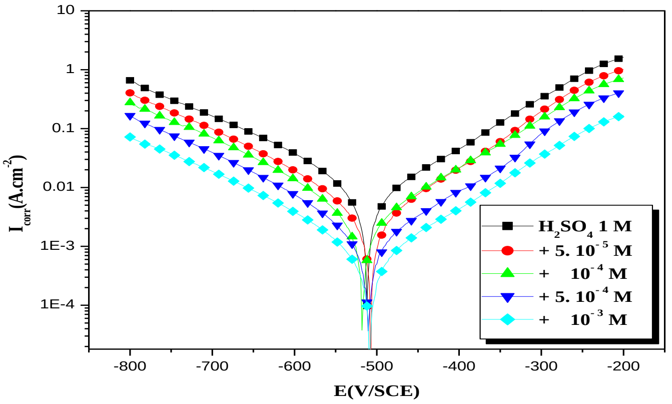

Fig. 5. Polarization curves for the system: mild steel/1M HCl containing 5-HOT

Table 2. Electrochemical parameters and corrosion inhibition efficiencies for the system: mild steel/5-HOT in different medium

	Conc. (Mol/L)	**E_{corr} (mV/SCE)**	**I_{corr} (mA/cm^2)**	**$-b_c$ (mV/dec)**	**$I.E_{Icorr}$ (%)**
1M HCl	Blank	-487	2.01	125	----
	5.10^{-5}	-498	0.77	127	**61.69**
	10^{-4}	-520	0.44	131	**78.10**
	5.10^{-4}	-534	0.32	141	**84.07**
	10^{-3}	-512	0.11	122	**94.52**
2M HCl	Blank	-475	12.30	161	----
	5.10^{-5}	-509	8.97	156	**27.07**
	10^{-4}	-513	5.81	153	**52.76**
	5.10^{-4}	-505	4.54	158	**63.08**
	10^{-3}	-511	2.50	150	**79.67**
0.5M H_2SO_4	Blank	-504	8.64	230	----
	5.10^{-5}	-485	5.84	232	**32.40**
	10^{-4}	-479	2.30	230	**73.37**
	5.10^{-4}	-487	1.14	231	**86.80**
	10^{-3}	-527	0.93	208	**90.08**
1M H_2SO_4	Blank	-510	13.46	180	----
	5.10^{-5}	-507	6.28	175	**53.34**
	10^{-4}	-514	3.41	180	**74.66**
	5.10^{-4}	-504	2.80	182	**79.19**
	10^{-3}	-512	1.37	181	**89.82**

Table 3. Polarization resistance and inhibition efficiencies for the system: mild steel/5-HOT in different medium

	Conc. (Mol/L)	R_p (Ω.cm^2)	$I.E_{Rp}$ (%)
HCl 1 M	Blank	13	-----
	5.10^{-5}	41	**68.28**
	10^{-4}	68	**80.88**
	5.10^{-4}	138	**90.56**
	10^{-3}	197	**93.40**
HCl 2 M	Blank	6.72	----
	5.10^{-5}	8.94	**33.03**
	10^{-4}	13.27	**46.36**
	5.10^{-4}	16.92	**60.28**
	10^{-3}	28.84	**76.70**
H_2SO_4 0.5M	Blank	3.52	---
	5.10^{-5}	5.56	**36.69**
	10^{-4}	11.82	**70.22**
	5.10^{-4}	16.94	**79.22**
	10^{-3}	25.71	**86.31**
H_2SO_4 1M	Blank	2.65	---
	5.10^{-5}	7.53	**64.81**
	10^{-4}	11.71	**77.37**
	5.10^{-4}	16.43	**83.87**
	10^{-3}	19.26	**86.24**

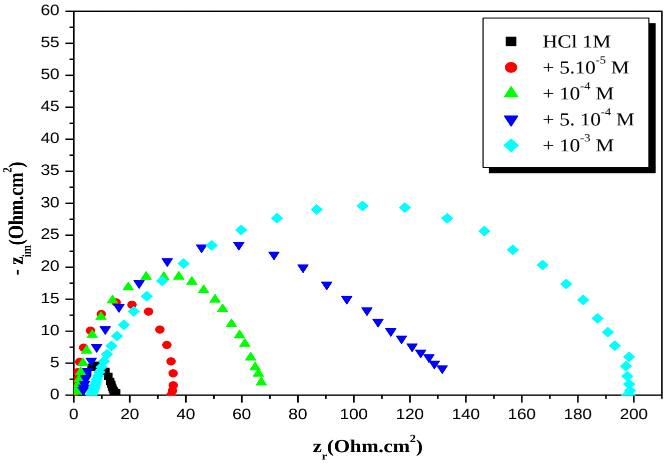

Fig. 6. Nyquist plots for mild steel in 1M HCl containing different concentrations of 5-HOT

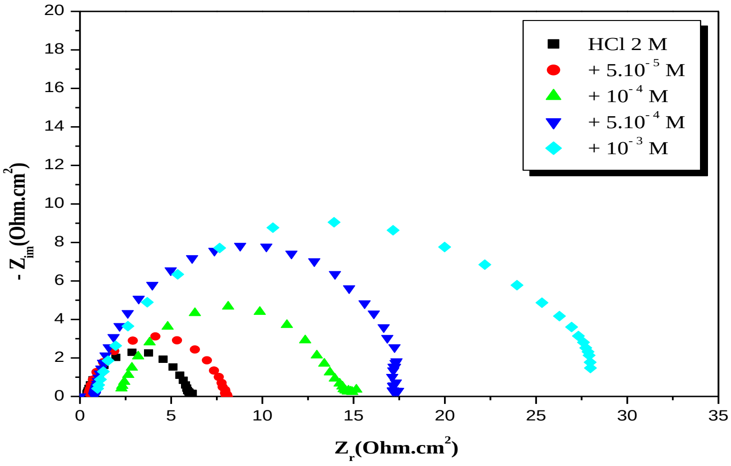

Fig. 7. Nyquist plots for mild steel in 1M HCl containing different concentrations of 5-HOT

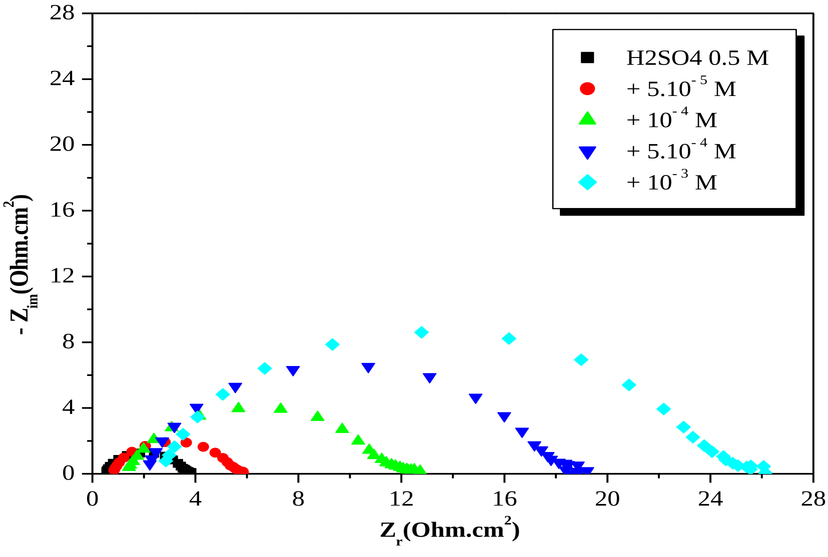

Fig. 8. Nyquist plots for mild steel in 1M HCl containing different concentrations of 5-HOT

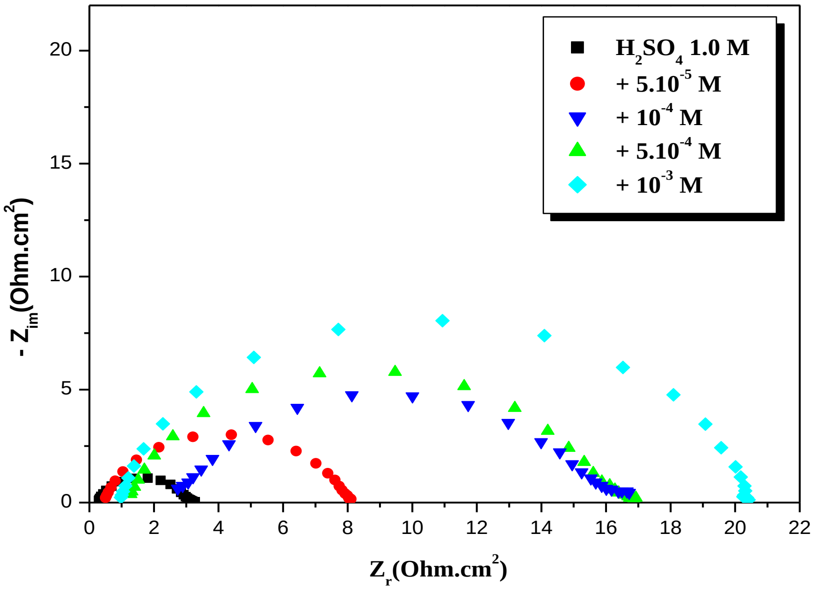

Fig. 9. Nyquist plots for mild steel in 1M HCl containing different concentrations of 5-HOT

Table 4. Impedance parameters and inhibition efficiencies for the system: mild steel/5-HOT in different medium

	Conc. (Mol/L)	**R_t (Ω.cm^2)**	**C_{dl} (μF/cm^2)**	**I.E$_{Rt}$ (%)**
HCl 1 M	Blank	12.5	82.67	----
	5.10^{-5}	39	102.02	**67.94**
	10^{-4}	70	56.84	**82.14**
	5.10^{-4}	140	45.47	**91.07**
	10^{-3}	200	39.78	**93.75**
HCl 2M	Blank	4.07	391.24	----
	5.10^{-5}	6.98	228.13	**20.34**
	10^{-4}	12.08	131.81	**56.05**
	5.10^{-4}	17.73	89.81	**62.81**
	10^{-3}	26.36	60.40	**76.99**
H_2SO_4 0.5M	Blank	2.58	246.87	---
	5.10^{-5}	5.71	176.50	**32.40**
	10^{-4}	12.69	125.48	**73.37**
	5.10^{-4}	14.18	112.29	**86.80**
	10^{-3}	16.17	98.47	**90.08**
H_2SO_4 1M	Blank	1.25	806.25	---
	5.10^{-5}	1.41	451.73	**61.10**
	10^{-4}	2.44	261.04	**76.5**
	5.10^{-4}	4.36	146.08	**82.37**
	10^{-3}	9.01	70.69	**85.15**

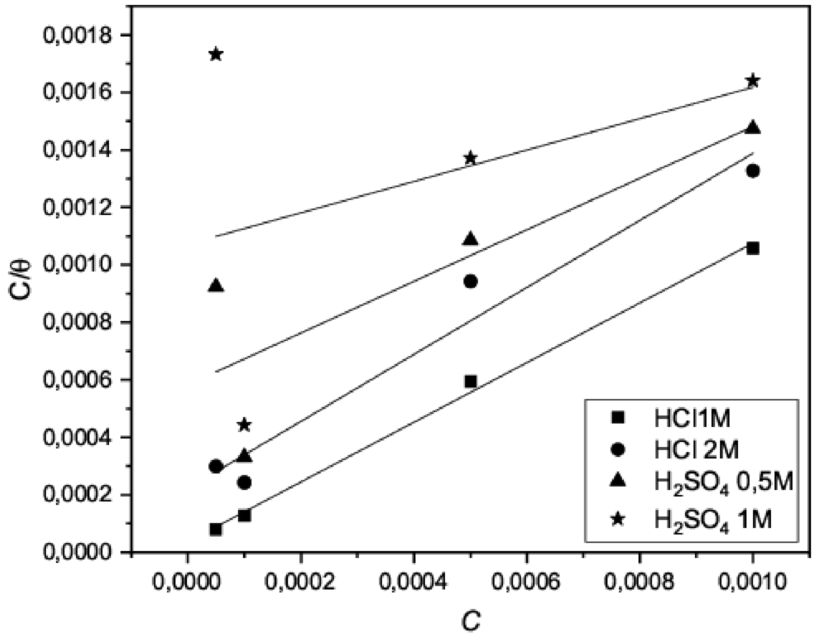

Fig. 11. Langmuir adsorption plots for the system: mild steel/different medium/5-HOT

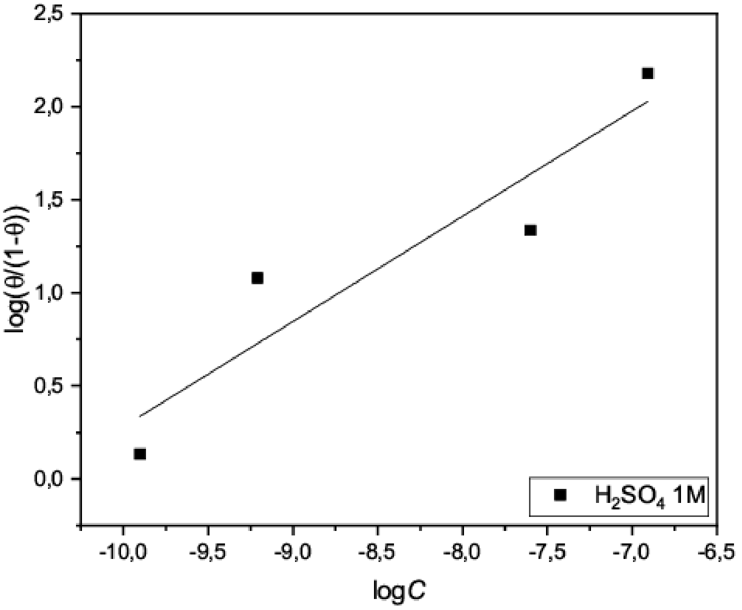

Fig. 12. El-Awady thermodynamic–kinetic model for the system: mild steel/1.5M H_2SO_4/5-HOT

Table 4. Thermodynamic parameters of adsorption obtained in different medium containing 5-HOT at 298 K.

Langmuir Isotherm			**kinetic–thermodynamic isotherm**		
1M HCl	K	$2.69x10^4$	1M H_2SO_4	lnK’	5.93
	ΔG_{ads}	**-34.49**		y	0.56
2M HCl	K	$3.66x10^4$		K_{ads}	24.01
	ΔG_{ads}	**-30.28**		$-\Delta G_{ads}$	**-17.82**
0.5M H_2SO_4	K	$1.71x10^4$			
	ΔG_{ads}	**-28.40**			

4. Conclusions

The tested compound (5-HOT) behaves as efficient inhibitor for the acidic corrosion of mild steel. The corrosion inhibition efficiency increased with increasing inhibitor concentration in different medium. The corrosion inhibition action of the 5-HOT is mainly due to its adsorption on the mild steel surface. The decrease in the corrosion rate of mild steel in the presence 5-HOT indicated that adsorbed protective layer formed on the steel surface is persistent different medium. The inhibitor under investigation was found to be mixed-type inhibitors, affecting both anodic and cathodic sites. Adsorption process obeyed Langmuir and El Awady kinetic-thermodynamic adsorption isotherm and regarded as physical adsorption with and little chemical adsorption for 1M HCl.

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