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Some azo derivatives as inhibitors for corrosion of C-steel in aqueous media

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Abstract

The inhibitive effect of 4-((4-formyl-3-hydroxyphenyl)diazenyl)-3-hydroxynaphthalene-1-sulfonic acid on carbon steel corrosion in hydrochloric acid solution was investigated using chemical and electrochemical techniques. It was found that the compound acts as good corrosion inhibitor with inhibition efficiency more than 98.5%. The inhibition efficiency was found to increase with inhibitor concentration and exposure time. On the other hand, it decreases by rising the temperature. The inhibition action was discussed in view of adsorption of the organic compound molecules on the carbon steel surface. It was found that the adsorption of 4-((4-formyl-3-hydroxyphenyl)diazenyl)-3-hydroxynaphthalene-1-sulfonic acid on carbon steel surface follows Langmuir adsorption isotherm. The thermodynamic parameters for the corrosion process in free and inhibited media were calculated and interpreted. Corrosion of metals and alloys is a disaster which causes a large economic loss in different human activities. The corrosion process is thermodynamically spontaneous and cannot be totally stopped anyhow. However, there are continuous trials to develop methods for decreasing the corrosion to its minimum rate. One of the methods used to achieve this goal was using inhibitors. The corrosion inhibitors act either by adsorption on the metal surface or reaction with it producing a passive film. The former ones are most likely organic compounds (Azo compounds) with atoms in their structures carrying lone pair of electrons capable of electrostatic attraction of the vacant orbitals of the metal.

Keywords: Carbon steel, Corrosion inhibitor, Electrochemical techniques, Thermodynamic parameters.
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1. Introduction

Modern steels are made with varying combinations of alloy metals to fulfill many purposes.[1] Carbon steel, accounts for 90% of steel production, composed simply of carbon and iron. [2] Low alloy steel is alloyed with other elements, usually molybdenum, manganese, chromium, or nickel, in amounts of up to 10% by weight to improve the hardenability of thick sections. High strength low alloy steel has small additions (usually < 2% by weight) of other elements, typically 1.5% manganese, to provide additional strength for a modest price increase[3].

Carbon steel is an important metal that is frequently used in industrial applications or household appliances. Although carbon steel forms a protective oxide layer on its surface, it suffers from corrosion if brought in contact with acidic media. This behavior is attributed to the formation of soluble salts by the reaction of the acid with the oxide layer. To avoid the corrosion of carbon steel in such cases inhibitor must be added to the cleaning acid. The inhibition of carbon steel corrosion in different media has been studied by many researchers either using organic and in organic compounds [4 -20].

Corrosion of metals and alloys is a disaster which causes a large economic loss in different human activities[21]. The corrosion process is thermodynamically spontaneous and cannot be totally stopped anyhow. However, there are continuous trials to develop methods for decreasing the corrosion to its minimum rate. One of the methods used to achieve this goal was using inhibitors. The corrosion inhibitors act either by adsorption on the metal surface or reaction with it producing a passive film. The former ones are most likely organic compounds with atoms in their structures carrying lone pair of electrons capable of electrostatic attraction of the vacant orbitals of the metal.

In the present work, 4-((4-formyl-3-hydroxyphenyl) diazenyl)-3-hydroxynaphthalene-1-sulfonic acid (Fig. 1) was investigated as inhibitors for carbon steel corrosion in 2.0 M HCl solution. 4-((4-formyl-3-hydroxyphenyl) diazenyl)-3hydroxynaphthalene-1-sulfonic acid has a formula $C_{17}H_{12}N_2O_6S$ with a molecular weight of 372.26464 g/mol. The chemical structure of 4-((4-formyl-3-hydroxyphenyl)diazenyl)-3-hydroxynaphthalene-1-sulfonic acid is represented in Fig.1. Weight loss and electrochemical techniques were used for this purpose. The thermodynamic parameters of the corrosion and inhibition processes were calculated. Moreover, the inhibition action of the tested compound was correlated with their structures using quantum computations.

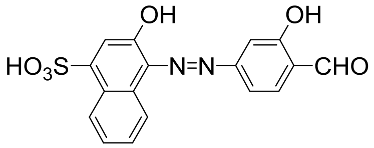

Fig (1): 4-((4-formyl-3-hydroxyphenyl) diazenyl)-3-hydroxynaphthalene-1-sulfonic acid

2. Experimental

Carbon steel sheets with dimension of (1.0 – 1.0 – 0.1 cm) were used for weight loss measurements. Prior to each experiment the surface of carbon steel specimens were mechanically polished with different grades of emery papers, degreased with acetone and rinsed by bi-distilled water, and dried between two filter papers. The cleaned carbon steel sheets were weighed before and after immersion in 50 ml of the test solution for a period up to 7days. The average weight loss for each two identical experiments was taken and expressed in mg/cm². The temperature was adjusted to 25±1°C using an air thermostat. Inhibition efficiencies were calculated from the weight loss data using the relation:

$$IE\% = \left(1-\frac{w_i}{w_f}\right) x\ 100 \qquad (1)$$

$$\theta = 1-\frac{w_i}{w_f} \qquad (2)$$

Where w_f and w_i are the rates of carbon steel coupon corrosion in free and inhibited HCl solutions, respectively. Three electrode cells with platinum foil as counter electrode and saturated calomel electrode was used in the electrochemical experiments. The working electrode was carbon steel rod impeded in araldite with bottom surface area of 1 cm^2 exposed to the corrosive solution. Before each experiment, the working electrode was polished with different grades of emery papers, washed by water and inserted in the electrochemical cell. The working electrode was immersed for 30 minutes in the test solution to attain its steady state potential before the start of each experiment. Meinsbergerpotentiostat/ Galvanostat with software zum PS6 remote, was used to carry out potentiodynamic polarization and cyclic voltammetry. Potentiodynamic experiments were performed by scanning the electrode potential from -1800 to 600 mV at 10mV/sec sweep rate. Tafel lines were extrapolated to the corrosion potential for the calculation of the electrochemical kinetics parameters. The IE% and θ were obtained by using equations (3), (4):

$$IE\% = \left(1-\frac{i_i}{i_f}\right) x\ 100 \qquad (3)$$

$$\theta = 1-\frac{i_i}{i_f} \qquad (4)$$

where i_f and i_i are the corrosion current densities of carbon steel specimen (mA/cm^2) in absence and presence of different concentrations of the inhibitor, respectively.

All experiments were carried out in normal aerated solutions at 30ºC except those carried out at different temperatures.

3. Results and discussion

3.1. Weight loss measurements

The corrosion behavior of carbon steel in 2.0 M HCl solutions devoid of and containing different concentrations of 4-((4-formyl-3-hydroxyphenyl) diazenyl)-3-hydroxy naphthalene-1-sulfonic acid was studied using weight loss technique at different exposure time intervals. The obtained data are tabulated in Table (1). The data of the table reveal that the addition of tested compound markedly decreases the corrosion of carbon steel in the acid solution. Therefore, it could be concluded that tested compound as good corrosion inhibitor for carbon steel corrosion in the acidic medium. The inhibition efficiency reaches to values more than 98.5%.

Table (1): Weight loss data of carbon steel corrosion in 2.0 M HCl solutions free of and containing different molar concentrations of tested compound at different exposure times. (r = corrosion rate; $mg.cm^{-2}.h^{-1}$)

t, h.		Free	0.00001	0.00005	0.0001	0.0005	0.001	0.005
24	*r*	0.004112	0.001291	0.001212	0.000739	0.000608	0.000477	0.000394
	IE%	-----	68.60	70.52	81.98	85.21	88.40	90.42
48	*r*	0.002723	0.000757	0.000669	0.000378	0.000351	0.000264	0.000206
	IE%	-----	72.20	75.43	86.12	87.11	90.30	92.43
72	*r*	0.002245	0.000552	0.000484	0.000259	0.000244	0.000190	0.000132
	IE%	-----	75.41	78.44	88.46	89.13	91.54	94.12
96	*r*	0.001933	0.000411	0.000351	0.000200	0.000165	0.000147	0.000091
	IE%	-----	78.74	81.84	89.65	91.46	92.40	95.30
120	*r*	0.001746	0.000323	0.000287	0.000171	0.000101	0.000088	0.000066
	IE%	-----	81.50	83.56	92.73	94.22	94.96	96.22
144	*r*	0.001591	0.000281	0.000199	0.000111	0.000077	0.000060	0.000042
	IE%	-----	82.34	87.50	93.02	95.10	96.23	97.42
168	*r*	0.001490	0.000232	0.000139	0.000087	0.000056	0.000040	0.000022
	IE%	-----	84.43	90.67	94.16	96.24	97.32	98.5

The inhibition action of 4-((4-formyl-3-hydroxyphenyl) diazenyl)-3-hydroxynaphthalene-1-sulfonic acid could be interpreted in view of its molecular structure. It is well known now that the adsorption of inhibitor molecules on the corroded metal surface is an essential initial step that should be established for performing the inhibition action.

The degree of adsorption of the chemical compound molecules largely depends on its chemical structure. The molecular structure of 4-((4-formyl-3-hydroxyphenyl) diazenyl)-3-hydroxynaphthalene-1-sulfonic acid contains two aromatic rings as well as O, N and S atoms which possess lone pairs of electrons. These features of the structure suggest a high tendency of the molecules to be strongly adsorbed at the metal surface. As the inhibitor molecules are adsorbed on the metal surface, they form a continuous film that acts as barrier between metal surface and the corrosive medium. The presence of this film prevents the transfer of mass and charge leading to the decrease of the corrosion process.

Fig (1) represents the relation between inhibition efficiency of 4-((4-formyl-3-hydroxyphenyl) diazenyl)-3-hydroxynaphthalene-1-sulfonic acid and its concentration for carbon steel corrosion in 2.0 M HCl solution at different time intervals. The curves of the figure show that, the inhibition efficiency increases as the inhibitor concentration is increased. It is important to note that the inhibition efficiency increases, from the lowest to the highest inhibitor concentration, in a narrow range; about one to two units. This behavior suggests that even the presence of low concentrations of tested compound leads to a high inhibition effect on the acid carbon steel corrosion. Such result may refer to the mode of inhibitor molecules adsorption on the metal surface. The high inhibition efficiency achieved by very small concentration is a fair evidence for horizontally adsorbed molecules. It implies that a small number of adsorbed molecules can cover a high surface area which could not be achieved if they are adsorbed vertically.

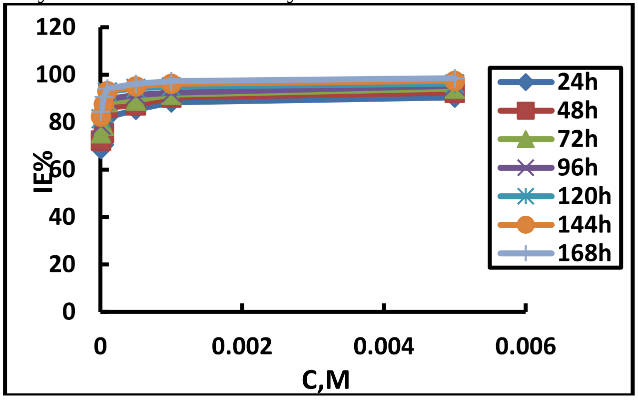

Fig (1): Effect of 4-((4-formyl-3-hydroxyphenyl) diazenyl) -3-hydroxynaphthalene-1-sulfonicacid concentration on inhibition efficiency for carbon steel corrosion in 2.0 M HCl solution.

Further examination of Figs (1) reveals that, the change of inhibition efficiency is relatively large upon increasing the inhibitor concentration from $5x10^{-5}$ to 5x 10^{-3} M. As the concentration reaches 5x 10^{-3} M, the inhibition efficiency steadily increases up to a concentration of $5x10^{-5}$ M. This result confirms the above conclusion regarding the adsorption mode of the inhibitor molecules. Thus, it takes no more than small number of molecules to approach the highest possible inhibition efficiency. Therefore, as such low inhibitor concentration 5x 10^{-3} M is reached, the surface is almost completely covered with adsorbed molecules. The small number of molecules needed for almost complete surface coverage supports the idea that the inhibitor molecules are adsorbed horizontally on the surface. Further increasing of inhibitor concentration only leads to avery small increase in the inhibition efficiency.

The little effect of increasing concentration on the inhibition efficiency could be interpreted as follows. At low inhibitor concentrations there are a lot of free sites on the metal surface for inhibitor molecules to adsorb at. Therefore, all the inhibitor molecules find their sites for adsorption and take their parts in the inhibition process. Nevertheless, as the concentration increases there are not enough free sites on the metal surface to all the molecules for adsorption. Thus, a competition between the inhibitor molecules arises to be adsorbed on the free surface site. Such competition for adsorption leads to decrease the impact of high concentration of the inhibitor molecules. The expected result of this is a deviation from linear relationship between inhibitor concentration and inhibition efficiency as shown in Fig. (1).

3.2.Polarization technique

The potentiodynamic polarization curves of carbon steel corrosion in 2.0 M HCl solutions in the absence and presence of various concentrations 4-((4-formyl-3-hydroxyphenyl)diazenyl)-3-hydroxynaphthalene-1-sulfonic acid were traced at scanning rate of 10 mV/sec. The obtained curves were represented in Fig. (2). The corrosion kinetic parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}), anodic Tafel slope (βa) and cathodicTafel slope (βc) deduced from the curves are given in Table (2).

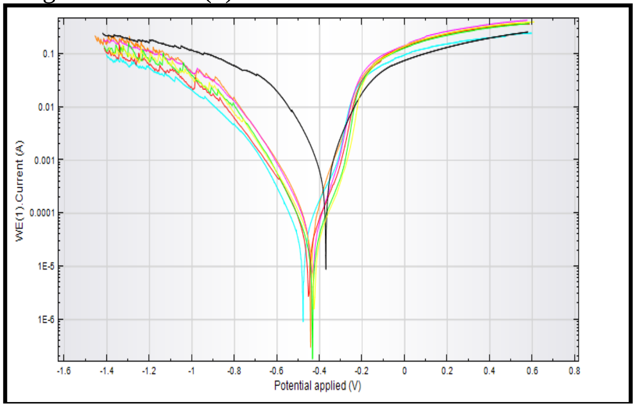

Fig (2): Potentiodynamic polarization of carbon steel in 2.0 M HCl solutions containing and devoid of different concentrations of 4-((4-formyl-3-hydroxyphenyl)diazenyl) -3-hydroxynaphthalene-1-sulfonic acid.

Inspection of Fig (2) reveals that both the anodic and cathodic polarization curves shift toward more negative potential and less current density values upon the addition of 4-((4-formyl-3-hydroxyphenyl)diazenyl)-3-hydroxynaphthalene-1sulfonic acid. This result suggests the inhibitive action of 4-((4-formyl-3-hydroxyphenyl) diazenyl)-3hydroxynaphthalene-1-sulfonic acid toward carbon steel corrosion in the acidic medium.Several observations, due to addition of the extract, could be recognized from the data of Table (2):

I. The corrosion potential tends to become more negative by the addition of 4-((4-formyl-3-hydroxyphenyl) diazenyl)-3-hydroxynaphthalene-1-sulfonic acid.
This result may refer to a cathodic inhibition mechanism.

II. The corrosion rate decreases, and the inhibition efficiency increases by increasing 4-((4-formyl-3-hydroxy phenyl) diazenyl)-3-hydroxynaphthalene-1-sulfonic acid concentration. Upon increasing the 4-((4-formyl-3-

hydroxyphenyl) diazenyl) -3-hydroxy naphthalene -1-sulfonic acid concentration in the bulk solution the number of molecules adsorbed on carbon steel surface increases leading to an increase in the inhibition efficiency.

Table (2): Parameters of carbon steel corrosion in free and inhibited 2 M HCl solutions as revealed from polarization technique.

Conc.,	- E_{corr},	βa	βc	i_{corr}	IE%
0.000000	0.37281	0.10112	0.12364	0.000505	-----
0.00001	0.42065	0.0846	0.173	9.15E-05	81.88
0.00005	0.37535	0.051943	0.22999	6.25E-05	87.62
0.0001	0.37786	0.052038	0.16976	4.64E-05	90.81
0.0005	0.37491	0.053015	0.18679	3.25E-05	93.56
0.001	0.34035	0.045797	0.18285	2.58E-05	94.36
0.005	0.3523	0.04285	0.16376	1.99E-05	96.06

There is almost change in anodic Tafel constant upon the addition of increasing concentrations of the 4-((4-formyl-3-hydroxyphenyl)diazenyl)-3hydroxynaphthalene-1-sulfonic acid. In contrast, the cathodic Tafel constant value has greatly changed upon the addition of the inhibitor. This result supports the conclusion made by the change of corrosion potential; that 4-((4-formyl-3-hydroxy phenyl)diazenyl)-3-hydroxynaphthalene-1-sulfonic acid acts as mixed inhibitor.

3.3. Adsorption behavior

The corrosion inhibition process is often attributed to the adsorption of inhibitor molecules onto the metal surface. The adsorbed molecules form a film isolating the metal surface from the aggressive medium. The best way to study the adsorption process is through identifying its adsorption isotherm. Several isotherms were postulated for different adsorption behaviors. All the known isotherms have been tried here in the present study to find out the one which is fitted with the obtained results. It is found that Langmuir adsorption isotherm is the best isotherm fitted with the experimental results.

Langmuir adsorption isotherm could be represented by the equation:

$$\frac{C}{\theta} = \frac{1}{k} + C \qquad (5)$$

Where C is the inhibitor concentration, θ is the fraction of surface coverage and k is the adsorption constant which is identified as:

$$lnk = ln\frac{1}{55.5} - \frac{\Delta G^{o}_{ads}}{RT} \qquad (6)$$

Where ΔG^{o}_{ads} *is* the standard adsorption free energy and 55.5 is the molar concentration of water.

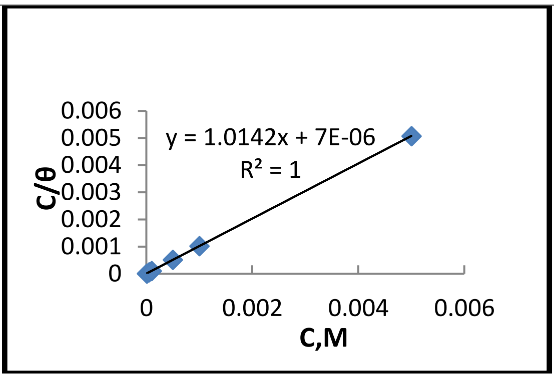

Fig (3): Langmuir adsorption isotherm

Plotting C versus C/θ gives a straight line with unit slope and unit correlation coefficient as shown in Fig. (3). This indicates that the adsorption 4-((4-formyl-3-hydroxy phenyl)diazenyl)-3-hydroxynaphthalene-1-sulfonic acid on carbon steel surface in the acidic medium follows Langmuir adsorption isotherm. The value of (k) was determined by the intercept of the line of Fig. (3) and was used to calculate the standard free energy of adsorption. It was found that standard free energy of adsorption of 4-((4-formyl-3-hydroxyphenyl)diazenyl)-3-hydroxy naphthalene-1-sulfonic acid on carbon steel surface in the acidic medium is -40.03 kJ/mol. The negative sign suggests that the adsorption process is spontaneous and forms a stable film on the carbon steel surface.

3.4. Effect of temperature

The effect of temperature (in the range of 313 – 343 K), on the corrosion of carbon steel in 2.0 M HCl solutions devoid of and containing $5x10^{-3}$ M of 4-((4-formyl-3-hydroxyphenyl)diazenyl)-3-hydroxynaphthalene-1-sulfonic acid was studied using Polarization technique**.** Table (3) contains the values of inhibition efficiency of $5x10^{-3}$M 4-((4-formyl-3-hydroxyphenyl) diazenyl) -3-hydroxynaphthalene-1-sulfonic acid toward carbon steel corrosion in 2.0 M HCl solutions at different temperatures for different exposure times. Inspection of Table (3) reveals that, as the temperature increases the inhibition efficiencies of all tested extract decrease.

Table (3): Corrosion parameters of carbon steel in 2 M HCl solutions devoid of and containing 0.005M of different extracts at different temperatures.

Temp. K	- E_{corr}, V	*βa* *V/decade*	*βc* *V/decade*	i_{corr} A/cm^2	*IE%*
303	0.37281	0.10112	0.12364	0.000505	--
313	0.36017	0.12196	0.16543	0.000586	
323	0.32345	0.088533	0.16988	0.000899	
333	0.463	0.063056	0.084328	0.001547	
343	0.49977	0.23712	0.27507	0.002501	
303	0.3523	0.04285	0.16376	1.99E-05	96.06
313	0.37786	0.052038	0.16976	4.64E-05	92.08
323	0.37922	0.0575	0.18954	7.31E-05	91.87
333	0.35438	0.22144	0.063731	0.000187	87.91
343	0.30268	0.087614	0.11769	0.000386	84.57

The corrosion reaction can be regarded as an Arrhenius-type process which follows the equation:

$$\log r = \log A - \frac{E_a}{2.303\,RT} \qquad (7)$$

Where (r) represents the rate of corrosion reaction, A is Arrhenius factor and E_a is the apparent activation energy of the corrosion reaction. Plotting of log r versus 1/T gave straight line, as shown in Fig(4). The values of activation energies for corrosion reactions of carbon steel in free and inhibited acid solutions were presented in Table (3).

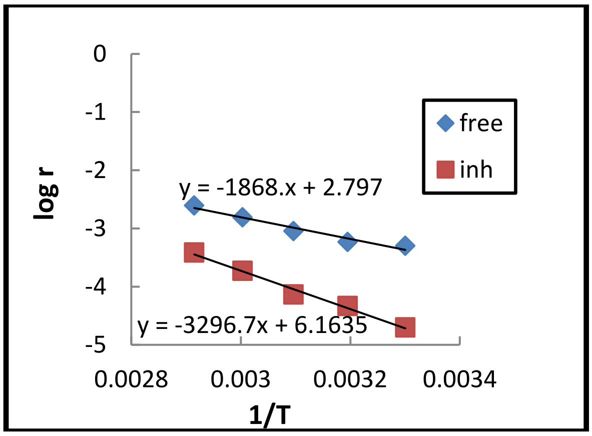

Fig (4): Arrhenius plot for carbon steel corrosion in the free and inhibited 2.0M HCl solutions.

Other activation parameters were calculated using the transition state equation:

$$\log \frac{r_{corr}}{T} = \left[\log\left(\frac{R}{hN}\right)\right] + \left[\frac{\Delta S^*}{2.303\,R}\right] - \frac{\Delta H^*}{2.303\,RT} \qquad (8)$$

where, R is the universal gas constant (8.314 J/mol.K), N is the Avogadro's number (6.02 x 10^{23}), h is the Plank's constant (6.62 × 10^{-34} m^2 kg /s) where ΔS^* and ΔH^* are the entropy and the enthalpy changes of activation corrosion energies for the transition state complex, respectively. Plotting log (r_{corr}/T) versus 1/T gives straight lines (Fig 5) from which the activation parameters are determined and represented in Table (4).

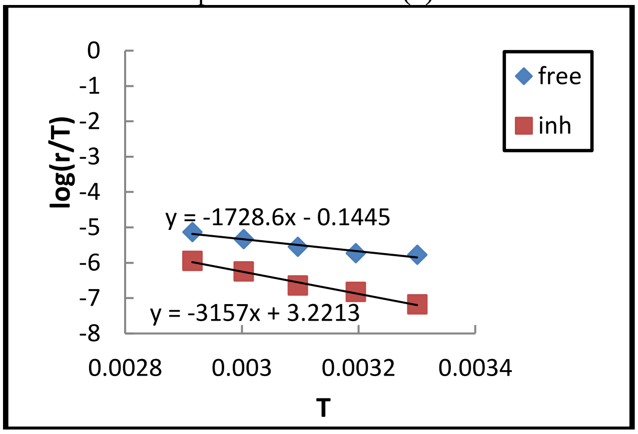

Fig (5): Transition state plot for carbon steel corrosion in free and inhibited 2.0M HCl solutions.

The data in Table (4) reveal that the presence of 4-((4-formyl-3-hydroxyphenyl)diazenyl)-3-hydroxynaphthalene-1-sulfonic acid increases the value of apparent activation energy of the corrosion process. The increase of the activation energy means a slowdown of the corrosion reaction.

Thus, 4-((4-formyl-3-hydroxyphenyl) diazenyl)-3-

hydroxynaphthalene-1-sulfonic acid acts as inhibitor for acid carbon steel corrosion via increasing the activation energy. This suggests that the molecules are adsorbed on the carbon steel surface forming a barrier for mass and charge transfer between the metal surface and the corrosive environment. Some authors[22]attributed such result to a physical adsorption process of the inhibitor molecules on the metal surface.

Table (4): Thermodynamic parameters of carbon steel corrosion reaction in free and inhibited 2.0M HCl solutions.

Medium	*E*a *kJ.mol^{-1}*	Δ*H** *kJ.mol^{-1}*	- Δ*S** *kJ.mol^{-1}K^{-1}*	Δ*G** (303 K) *kJ.mol^{-1}*
Free	35.77	33.09	0.195	92.18
Inhibited	63.41	60.45	0.136	101.66

The change in the free energy of activation (ΔG*) for the corrosion process was calculated at 303K by applying the well-known thermodynamic equation:

$$\Delta G^* = \Delta H^* - T\,\Delta S^*$$

The obtained ΔG* values were also listed in Table (4). According to the data recorded in Table (4) ΔH* has a positive sign, reflecting the endothermic nature of the activation process, for the corrosion reaction. The negative values of ΔS* point out to a greater order produced during the process of activation. This can be achieved by the association or fixation process associated with the formation of activated complex with consequent loss in the degrees of freedom of the system during the process [23].The values of ΔG* were positive indicating that the activated complex is not stable. However, ΔG* values for the inhibited systems were somewhat more positive than that for the uninhibited systems. This result reveals that, in the inhibited acid solution the activated complex becomes less stable as compared with that in the free acid one. Thus, the presence of inhibitor decreases the probability of activation complex formation.

4. Conclusions

It was found that 4-((4-formyl-3-hydroxyphenyl) diazenyl)-3-hydroxynaphthalene-1-sulfonic acid acts as good corrosion inhibitor with inhibition efficiency more than 98.5%. The inhibition efficiency was found to increase with inhibitor concentration and the exposure time but decreases by rising the temperature. The adsorption of 4-((4-formyl-3-hydroxyphenyl)diazenyl)-3-hydroxynaphthalene-1-sulfonic acid on carbon steel surface follows Langmuir adsorption isotherm. The calculated thermodynamic parameters for the corrosion process in free and inhibited media show that the corrosion process is endothermic and the transition complex is less stable in the presence of 4-((4-formyl-3-hydroxy phenyl)diazenyl)-3-hydroxynaphthalene-1-sulfonic acid. Moreover, the activation energy increased in the presence of 4-((4-formyl-3-hydroxyphenyl)diazenyl)-3-hydroxy naphthalene-1-sulfonic acid.

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