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THERMODYNAMIC PROPERTIES OF SALICYLIC ACID AS CORROSION INHIBITOR OF CARBON STEEL IN HYDROCHLORIC ACID PICKLING ENVIRONMENT

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ABSTRACT

The corrosion inhibition of carbon steel in 0.1 M HCl by salicylic acid (SA) was investigated using weight loss method in the temperature range of 313-343 K. It was found that the inhibition efficiency increases with an increase in (SA) concentration but decreases with an increase in temperature. It has been determined that the adsorption for the studied inhibitor on carbon steel complies with the Langmuir adsorption isotherm at all studied temperatures. Both kinetic parameters (activation energy, pre-exponential factor, enthalpy of activation and entropy of activation) and thermodynamics of adsorption (enthalpy of adsorption, entropy of adsorption and Gibbs free energy) were calculated and discussed.

KEYWORDS: salicylic acid, Carbon steel, Hydrochloric Acid, Corrosion inhibition, Thermodynamic, Adsorption.

1. INTRODUCTION

Carbon steel is a common constructional material for many industrial units because of its low cost and excellent mechanical properties. However, it suffers severe attack in service particularly in oil and gas production systems. Although corrosion inhibitors are the most effective and flexible mean of corrosion control in oil and gas production systems, the selection and application of inhibitors are actually complicated because of the variable corrosive environments in these systems.[1] Especially in environment containing Cl⁻ ions, corrosion of metals is unavoidable. As Cl⁻ ion is an active one, it forces oxide formation on metal surface by adsorbing as alternative on metal surface. This increases the corrosion rate of metal.[2] The use of inhibitors is one of the most practical methods for protection against corrosion in acidic media. Most well-known acid inhibitors are organic compounds, such as those containing nitrogen, sulphur, an aromatic ring and oxygen atoms.[3–12] The selection of appropriate inhibitors mainly depends on the type of acid, its concentration, temperature, the presence of dissolved inorganic and/or organic substances even in minor amounts and, of course, on the type of metallic material exposed to the action of acidic solution.[13] Generally speaking, corrosion inhibitors are found to protect steel corrosion in acid solutions by adsorbing themselves on

steel surface. Adsorption is a separation process involving two phases between which certain components can become differentially distributed.

Adsorption can be described by two main types of interaction[14]

• Physisorption, involves electrostatic forces between ionic charges or dipoles on the adsorbed species and the electric charge at the metal/solution interface. The heat of adsorption is low and therefore this type of adsorption is stable only at relatively low temperatures.

• Chemisorption, involves charge sharing or charge transfer from the inhibitor molecules to the metal surface to form a coordinate type bond. In fact, electron transfer is typically for transition metals having vacant low-energy electron orbital. Chemisorption is typified by a much stronger adsorption energy than physical adsorption. Such a bond is therefore more stable at higher temperatures.

• Thermodynamic adsorption parameters and kinetic corrosion parameters are a useful tool for clarifying the adsorption behaviour of an inhibitor. Earlier work for Riggs and Hurd.[15] revealed that from a comparison of activation energies of uninhibited and inhibited corrosion reaction, heats of inhibitor adsorption may obtain. However, it was found that while positive heat of adsorption, $\Delta H_{ads} > 0$ (endothermic process) is

attributed unequivocally to chemisorption[24], a negative heat of adsorption, ΔHads < 0 (exothermic process) may involve either physisorption or chemisorption[16] or mixture of both processes (comprehensive adsorption).[17]

The purpose of the present work is to investigate the effect of salicylic acid (SA) as corrosion inhibitor for carbon steel in 1M HCl at various temperatures using gravimetric measurements. The kinetic and adsorption parameters of corrosion inhibition process are also evaluated and is cussed.

2. Experimental
2.1. Materials And Apparatus
salicylic acid (SA), the steel used in this study is a carbon steel (ST37) with a chemical composition (in wt%) of 0.08 % C, 0.05 % S, 0.25 % Mn, 0.04 % P, and the remainder iron (Fe). The carbon steel samples were pre-treated prior to the experiments by grinding with emery paper SiC (80, 180, 400, 600 and 1200); rinsed with distilled water, again with bidistilled water and then dried at room temperature before use. The acid solutions (0.1 M HCl) were prepared by dilution of an analytical reagent grade 37 % HCl with double-distilled water. The concentration range of salicylic acid (SA) employed was 0.01-0.025 M.

2.2. Weight loss measurements
The steel sheets of 4.2 cm x 2 cm x 0.1 cm dimensions were abraded with different grades of emery papers, washed with distilled water, degreased with acetone, dried and kept in a desiccator. After weighing accurately by a digital balance with high sensitivity the specimens were immersed in solution containing 0.1 M HCl solution with and without various concentrations of the investigated inhibitor. At the end of the tests, the specimens were taken out, washed carefully in ethanol and then dried, weighed accurately. The immersion time for the weight loss is 6h. Weight loss allowed calculation of the mean corrosion rate in (mg.cm-2.h-1). The corrosion rate (CR) and the inhibition efficiency (IE%) were calculated by the following equations:

$$CR = \frac{w_0 - w}{A.t} \qquad (1)$$

$$IE\% = \frac{CR_0 - CR}{CR_0} * 100 \qquad (2)$$

The degree of surface coverage for different concentrations of inhibitor is calculated using:

$$\theta = \frac{CR_0 - CR}{CR_0} = \frac{IE\%}{100} \qquad (3)$$

where w0 and w are the weights of carbon steel sheet before and after experiments A is the total surface area of

the specimen, t is the immersion time and CR0 and CR are values of the corrosion rate without and with addition of the inhibitor, respectively.

3. RESULTS AND DISCUSSION
3.1. Effect of temperature and kinetic parameters of activation
The influence of temperature on the corrosion behaviour of steel/acid in the presence and absence of the (SA) at various concentrations is investigated by weight-loss trend in the temperature rang 313-343 °K during 6h of immersion. For this purpose, gravimetric experiments were performed at different temperatures and the results are given in Table 1. Table 1 indicates that at a given (SA) concentration the corrosion rate of carbon steel increased with temperature. The increase is more pronounced at low concentrations. The results also indicate that for a given temperature, the corrosion rate of carbon steel decreased with increasing inhibitor concentration. Is clear that inhibition efficiency increased with increase in inhibitor concentration. The maximum value of inhibition efficiency (IE%) obtained for 0.025 M is 96% at 313 K. It shows that inhibition efficiency decreased at higher temperatures. This behaviour indicates desorption of inhibitor molecule.[18]

The activation parameters for the corrosion process were calculated from the Arrhenius equation[19]

$$CR = AExp(\frac{-Ea}{RT}), \quad Ln(CR) = LnA - \frac{Ea}{RT} \qquad (4)$$

where Ea represents the apparent activation energy, R is the molar gas constant (8.31434 J.K^{-1}.mol^{-1}), and A is the frequency factor. Arrhenius plots for the corrosion rate of carbon steel are given in Figure 1. Values of Ea for carbon steel corrosion in 0.1 M HCl in the absence and presence of (SA) were calculated by linear regression between Ln (CR) and 1/T and are listed in Table 2. An alternative form of Arrhenius equation is also used to calculate enthalpy and entropy of activation.[19]

$$CR = \frac{RT}{Nh} Exp(\frac{\Delta S_a}{R})Exp(\frac{-\Delta H_a}{RT}) \qquad (5)$$

$$Ln(\frac{CR}{T}) = Ln(\frac{R}{Nh}) + \frac{\Delta S_a}{R} - \frac{-\Delta H_a}{RT}$$

where CR is the corrosion rate, h is the Planck's constant (6.626176 x 10-34 Js), N is the Avogadro's number (6.02252 x 10 mol-1), R is the universal gas constant and T is the absolute temperature, ΔHa the enthalpy of activation, and ΔSa entropy of activation. The values of enthalpy of activation ΔHa and entropy of activation, ΔSa were obtained from the transition state equation.[5] A plot of Ln (CR /T) as a function of 1/T (Fig. 2) was made for carbon steel corrosion in 0.1 M HCl in the absence and presence of different concentrations of (SA). Straight lines are obtained with a slope (-ΔHa /R) and intercept (Ln(R/Nh) + ΔSa /R) from which the ΔHa and ΔSa values are calculated (Table 2).

Table 1: The values of inhibition efficiency obtained from the weight loss for SA at different concentration in 0.1 M HCl and at different temperatures.

SA Conc (M)	Temp. (°K)	CR (mg.cm^{-2}.h^{-1})	IE (%)	θ
0	313	0.7602	0	0
	333	2.3815	0	0
	343	4.1509	0	0
0.01	313	0.1889	75.1523	0.7515
	333	0.5620	76.3997	0.7640
	343	1.0815	73.9460	0.7395
0.015	313	0.1333	82.4604	0.8246
	333	0.4315	81.8818	0.8188
	343	0.7546	81.8202	0.8182
0.02	313	0.1037	86.3581	0.8636
	333	0.3269	86.2753	0.8628
	343	0.6428	84.5153	0.8451
0.025	313	0.0306	95.9805	0.9598
	333	0.1259	94.7123	0.9471
	343	0.2513	93.9462	0.9395
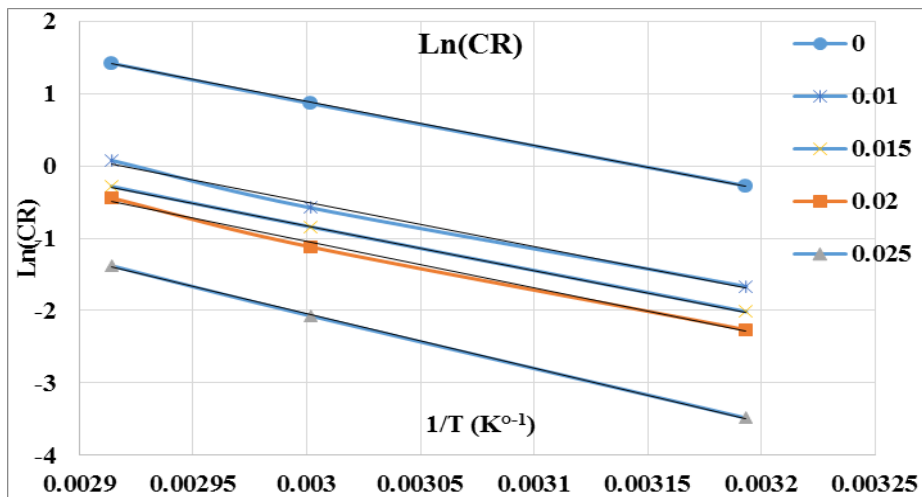
Fig. 1. Arrhenius plots of carbon steel in acid with and without different concentrations of SA.
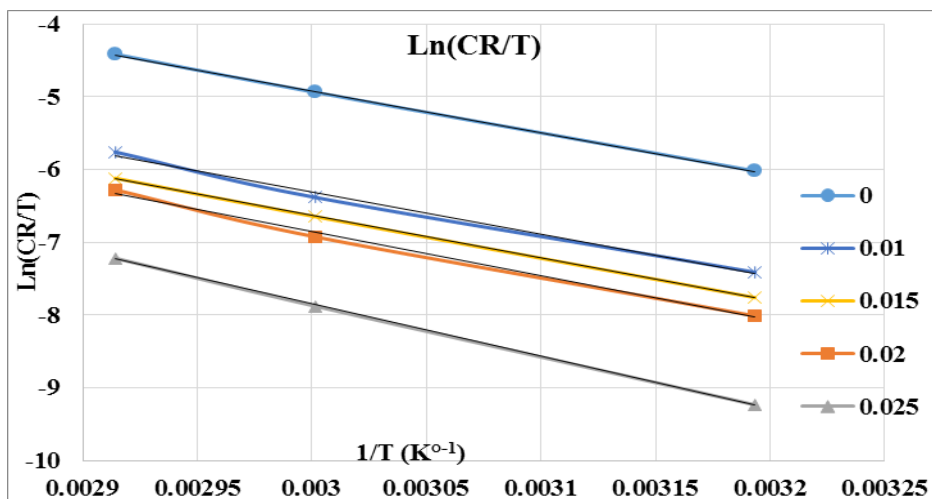
Fig. 2: Transition-state plots for carbon steel corrosion rates Ln (CR /T) versus 1/T in 0.1 M HCl in absence and in presence of different concentrations of SA.

The values of activation energy were obtained from the slopes of the linear plots and are given in Table 2. These values indicate that the presence of SA increases the activation energy of the metal dissolution reaction. The

adsorption of the inhibitor is assumed to occur on the higher energy sites and the presence of the inhibitor, which results in the blocking of the active sites, must be associated with an increase in the activation energy of carbon steel corrosion in the inhibited state.[20] The higher value of Ea in the presence of SA compared to that in its absence and the decrease in the IE (%) with

rise in temperature is interpreted as an indication of physisorption.[20-22]

As a result, the increase of SA concentration leads to an increase in the value of Ea, indicating strong adsorption of the inhibitor molecules at the metal surface.

Table 2: Activation parameters of the dissolution of carbon steel in 0.1 M HCl in the absence and presence of different concentrations of SA.

| Conc. (M)	E_a (kJ/mol)	ΔH_a (kJ/mol)	ΔS_a (J/mol K)	$E_a - \Delta H_a$ (kJ/mol)
0	-50.38	-47.6658	-95.42	-2.718
0.01	-51.20	-48.48	-104.53	-2.718
0.015	-51.51	-48.79	-106.29	-2.719
0.02	-53.58	-50.87	-101.90	-2.718
0.025	-62.53	-59.81	-63.43	-2.718

Examination of these data reveals that the ΔHa values for dissolution reaction of carbon steel in 0.1 M HCl in the presence of SA are higher than that in the absence of SA. The positive sign of ΔHa show the endothermic nature of the solution process suggesting that the dissolution of carbon steel is slow, which indicates that inhibition efficiencies decrease with increase in temperature. From Table 2, it is seen that the value of activation energy and enthalpy of activation varied in the same way. This result verified the known thermodynamic relation between Ea and ΔHa.[23,24]

$$E - \Delta H = RT \qquad (6)$$

The large negative value of ΔSa for carbon steel in 0.1 M HCl implies that the activated complex is the rate-determining step, rather than the dissociation step. In the presence of the inhibitor, the values of ΔSa increases and is generally interpreted as an increase in disorder as the reactants are converted to the activated complexes.

3.2. Adsorption isotherm and thermodynamic consideration
Adsorption isotherms provide information about the interaction of the adsorbed molecules with the metal surface. The adsorption of organic compounds can be expressed by two main types of interactions: physical adsorption and chemical adsorption. There are some factors that influence the adsorption processes including the nature and charge of metal, the chemical of inhibitor, and the type of electrolyte.[25] The adsorption of an organic adsorbate at metal-solution interface can be presented as a substitution adsorption process between the organic molecules in aqueous solution, Org(sol), and the water molecules on metallic surface, H2O (ads).[26]

$$Org(sol) + nH_2O(ads) \rightarrow Org(ads) + nH_2O(sol)$$
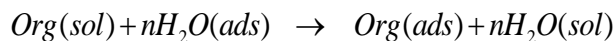
where Org(sol) and Org(ads) are the organic specie dissolved in the aqueous solution and adsorbed onto the metallic surface, respectively, H2O(ads) is the water molecule adsorbed on the metallic surface and n is the size ratio representing the number of water molecules

replaced by one organic adsorbate. For the studied inhibitor, it was found that the experimental data obtained from polarization readings could be fitted by Langmuir's adsorption isotherm.

According to this isotherm, the surface coverage θ is related to inhibitor concentration by.[27, 28]

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \qquad (7)$$

where, Kads is adsorption equilibrium constant, Cinh is the inhibitor concentration and θ is the surface coverage that was calculated by Eq. (3). Plots of (Cinh/θ) versus Cinh yield a straight line as shown in Fig. 3. In both cases the linear regression coefficients (R2) are almost equal to 1 and the slopes are very close to 1, indicating that the adsorption of SA in 0.1 M HCl follows the Langmuir isotherm and exhibit single-layer adsorption characteristic. The equilibrium adsorption constant, Kads, for the adsorption of the compound at different temperature were calculated from the slopes of straight lines and listed in Table 3.

It is seen from the Figure (3) that the correlation coefficients are very good and Kads values decrease with an increase in temperature. Large values of Kads mean better inhibition efficiency of the inhibitor, i.e., strong electrical interaction between the double-layer existing at the phase boundary and the adsorbing inhibitor molecules.[29]

Kads is related to the free energy of adsorption ΔG°ads by the equation

$$K_{ads} = \frac{1}{55.5} Exp(\frac{-\Delta G^{\circ}_{ads}}{RT}) \qquad (8)$$

where 55.5 is the concentration of water expressed in mol/L (the same as that of inhibitor concentration). The calculated ΔG°ads values, at all studied temperatures, are given in Table 3.
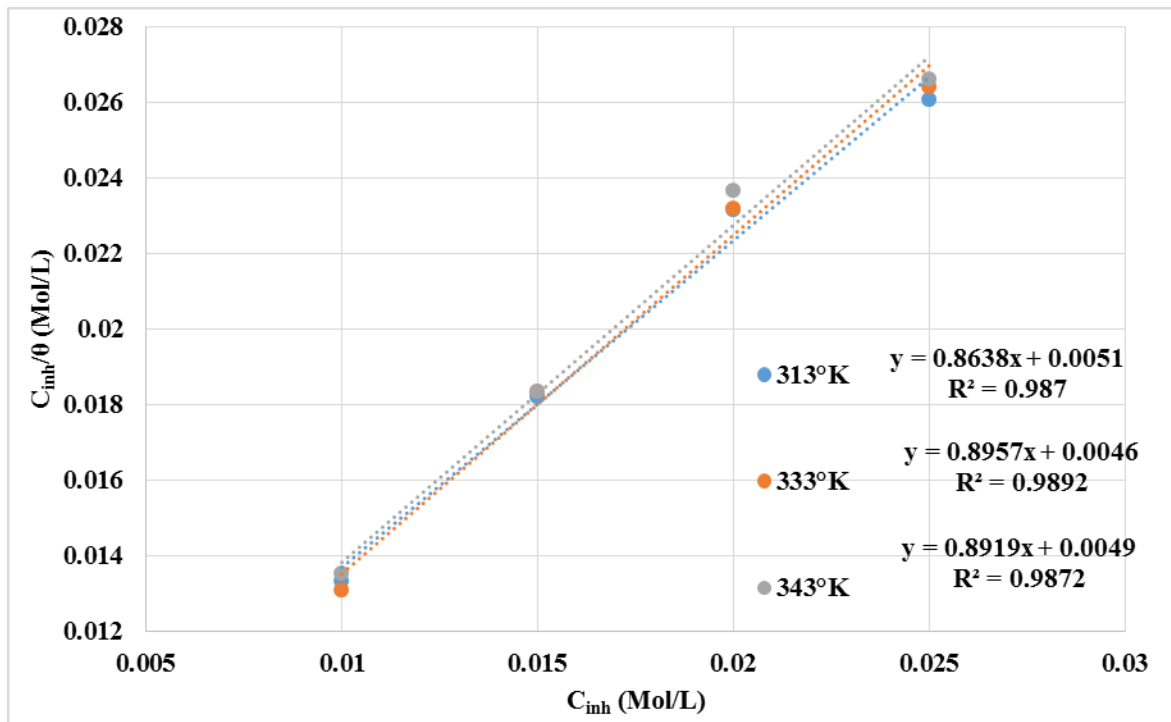
Fig. 3: Langmuir's isotherm adsorption model of SA on the carbon steel surface in 0.1 M HCl at different temperatures.

Table 3: The thermodynamic parameters of adsorption of SA on the carbon steel surface.

T (°K)	K_{ads} (L/Mol)	ΔG°_{ads} (KJ/Mol)	ΔH°_{ads} (kJ/mol)	ΔS°_{ads} (J/mol)
313	196.0784	-24.1995		
333	217.3913	-26.0309	-1.727	82.9204
343	204.0816	-26.632		

Generally, values of ΔG°_{ads} up to -20 kJ mol-1 are consistent with electrostatic interaction between charged molecules and a charged metal (which indicates physical adsorption) while those more negative than -40 kJ mol-1 involves charge sharing or transfer from the inhibitor molecules to the metal surface to form a coordinate type of bond (which indicates chemisorption).[30] The calculated standard free energy of adsorption values in the studied temperature domain are in the range of -24 to -27 kJ mol-1. therefore, it can be concluded that the adsorption is a physical adsorption.[31,32] The enthalpy and entropy of adsorption (ΔH°_{ads} and ΔS°_{ads}) can be calculated using the following equations.[33]

$$\Delta G^{\circ}_{ads} = \Delta H^{\circ}_{ads} - T\Delta S^{\circ}_{ads} \qquad (9)$$

$$LnK_{ads} = -\frac{\Delta H^{\circ}_{ads}}{RT} + \frac{\Delta S^{\circ}_{ads}}{R} - Ln(55.5) \qquad (10)$$

Fig. 4 represents the plots of Ln (Kads) versus (1/T) for adsorption SA.

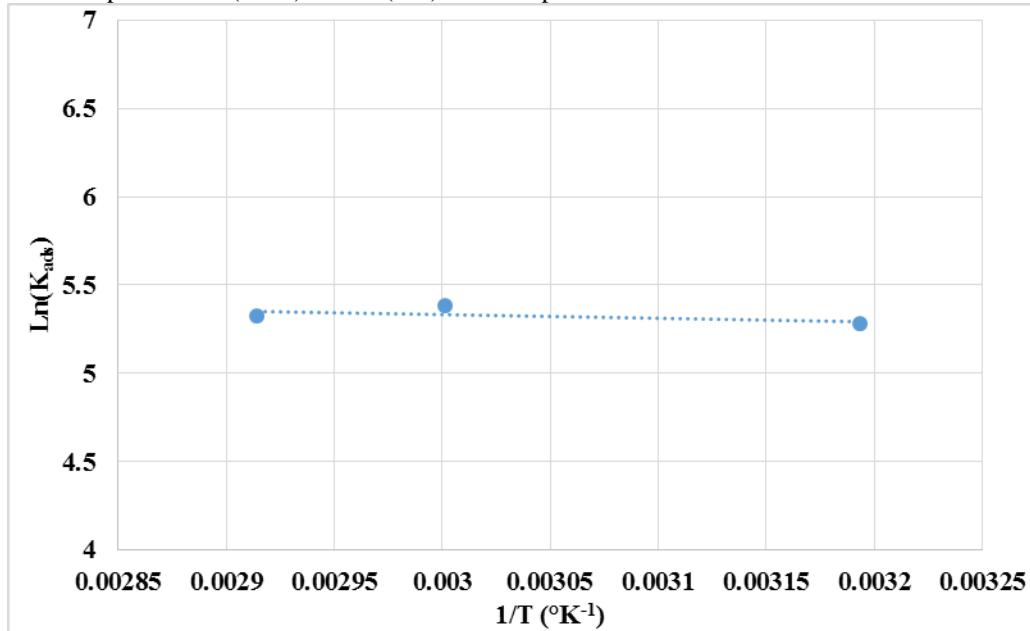**Fig.4: The relationship between Ln (Kads) and 1/T for SA.**

The lines obtained represent a slope of ($-\Delta H^{\circ}_{ads}$ /R) and intercept of [-Ln (55.5) + ΔS°_{ads} /R]. The values of Kads and ΔG°_{ads} are listed in Table 3. The calculated values of ΔH°_{ads} and ΔS°_{ads} are -1.727 kJ.mol-1 and 82.9204 J.mol^{-1}.K^{-1} respectively.

An endothermic adsorption process ($\Delta H^{\circ}_{ads} > 0$) is due to chemisorption while an exothermic adsorption process ($\Delta H^{\circ}_{ads} < 0$) may be attributed to physisorption, chemisorption or a mixture of both.

When the process of adsorption is exothermic, physisorption can be distinguished from chemisorptions according to the absolute value of ΔH°_{ads}. For physisorption processes, this is usually lower than -40 kJ mol^{-1} while its value is around -100 kJ mol^{-1} for chemisorptions.

Base on the results of the present work, the calculated ΔG°_{ads} and ΔH°_{ads} values for inhibitor show that adsorption mechanism is completely physical and a physisorption exists between the inhibitor and metal surface.

The adsorption process of SA onto steel surface includes, at least, two steps: the adsorption of SA molecules onto steel surface and simultaneous desorption of some hydration water from the substrate surface. The former process is expected to cause a decrease in randomness, thus in ΔS°_{ads}. The later process could cause some increase in disorderness. The obtained entropy value seems to be in contrast to that normally accepted in the adsorption phenomenon. While the adsorption of organic molecules is generally exothermic with decrease in entropy, the contrary occurs for the desorption of water molecules. The thermodynamic values obtained are the algebraic sum of those of the adsorption of organic molecules and desorption of water molecules. Therefore, the positive value of ΔS°_{ads} related to substitutional adsorption can be attributed to the increase in the solvent entropy and to more positive water desorption entropy.[34] The positive value of ΔS°_{ads} was also interpreted by the increase of disorder due to desorption of more water molecules from the metal surface by one inhibitor molecule.[35]

4. CONCLUSION
The salicylic acid (SA), showed good corrosion inhibition performance for carbon steel in 0.1 M HCl. The inhibition efficiency increases with increase in the concentration of SA but decreases with increase in temperature. Kinetic parameters were evaluated and discussed. The adsorption of SA on the carbon steel surface in 0.1 M HCl obeyed the Langmuir adsorption isotherm model at all studied temperatures. The values of ΔG°_{ads} are negative which suggest that the molecules of this inhibitor were strongly adsorbed on the carbon steel surface. The values obtained support the physical adsorption mechanism.

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