

Study of the Behavior of Sodium Diclofenac Expired Drug as an Corrosion Inhibitor of Iron in Sulfuric Acid Using UV-VIS Spectrophotometry

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ABSTRACT

The effect of *Sodium Diclofenac* (SD) as an inhibitor of pure iron corrosion in a 1.0 N of sulfuric acid was studied spectrally using a UV-VIS at different temperatures of 343.15, 323.15, 303.15 K. The concentration of dissolved iron was determined spectrally using phenanthroline method. The absorbance of each solution was measured at different times, and the data obtained were used to calculate the corrosion rate, which was found to be a zero-order reaction in the presence and absence of the SD inhibitor. The absorbance was decreased with an increase in inhibitor concentration and an increase in temperature. However, the inhibition efficiency was increased with inhibitor concentration an increase. The inhibitor is adsorbed on the surface of the iron particles according to the Langmuir and Kinetic-thermodynamic model of adsorption isotherm. The results indicated that the reaction is endothermic and unspontaneous. Quantum chemical parameters such as the highest & lowest occupied molecular orbital energy (E_{HOMO}) & (E_{LUMO}), energy gap (ΔE), were calculated and correlated to the experimental data.

Keywords: Iron, Inhibitor, Expired Drug, Sodium Diclofenac, Spectrophotometry, Adsorption Isotherm

I. INTRODUCTION

Corrosion is the main hazard problem for the petroleum industry. Generally, storage tanks, pipelines, heat exchangers, phase separators, and other industrial equipment gradually corroded by chemical or electrochemical reactions within their environment [1]. The most common form of corrosion in the oil and gas industry occurs when steel comes in contact with an aqueous environment [2]. Corrosion monitoring is the checking of the destruction of materials over time, and its goal is to track potential problem areas in equipment so that accidents are avoided. There are several different

non-destructive detection techniques such as ultrasonic and acoustic methods, an electromagnetic method, radiographic method. Whoever, destructive methods are coupons and probes like electrical resistance, electrochemical impedance spectroscopy and linear polarized resistance [3]. However, these techniques often result in expensive and time-consuming to clear and inspect the tank, which can influence the normal production[4].

A colorimetric method was used by Johnsson [5], to investigate the corrosion rates of steels by measuring the concentration of dissolved iron in an aqueous phase [6]. As reagent, ortho-phenanthroline was used

which reacted instantaneously with the divalent Fe forming a colored complex of $[\text{Fe}(\text{C}_{12}\text{H}_8\text{N}_2)_3]^{+2}$ [6]. Advantages of the use of 1,10-phenanthroline in the determination of iron may be summarized as follows [7, 8]: (1) it gives a relatively more intense colour for a given iron concentration the orange-red complex produced is stable, and no fading has been observed for as long as six months; (2) pH need not be regulated closely; (3) colour formations occur in acid solution, eliminating the difficulties usually caused by precipitation of metal hydroxides and hydrated oxides in alkaline solution, (4) the method is most sensitive for concentrations of about 1 to 3 ppm In the strongly acid solution the remaining from of iron is ferric ions. Thus, reduction the ferric to the ferrous condition by hydroxylamine hydrochloride before the addition of the 1,10-phenanthroline is necessary [7]. Since the method can be used to determine iron in concentrations as low as 0.5 ppm, it can advantageously be adapted to the determination of iron in the food and biological materials and analytical reagents[7]. It is now accepted that the spectrophotometer provides a quantitative analysis that is of a higher degree of accuracy than other methods.

In light of the above observation and related to our previous work [11-14], we aimed to investigate the corrosion inhibition properties of expired drug namely *Sodium Diclofenac* "Fig. 1" on pure iron in 1.0 N H_2SO_4 solution utilizing the spectroscopic method. *Sodium Diclofenac*, a drug used was selected as a corrosion inhibitor based on its molecular structure that has O, N active center atoms. There is a big concern in replacing environmental unsafe inhibitors with effective non-toxic alternatives. Expired drugs (non-toxic characteristics) were used as corrosion inhibitors for metal alloys. Most of the chemical drugs are more expensive than the organic inhibitors used in industries. Thus, expired drugs that are of no use can be investigated as corrosion inhibition. In this way, the environmental and economic problems concern the pollution with a pharmaceutically active

expired drug that can be solved [15]. The visible spectroscopic technique was used for this purpose is very simple and gives excellent results for the detection of trace amounts of iron.

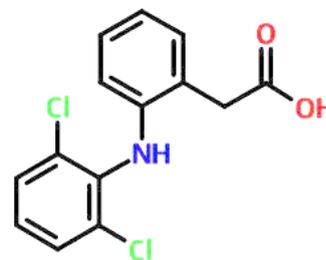


Figure 1: Inhibitor molecule of *Sodium Diclofenac* (SD) drug

II. METHODS AND MATERIAL

A. Chemicals and Instrumentation

All reagents were of analytical grade. Deionized water was used throughout. All the tests were performed on the pure iron powder from Lab Chemicals, 99%, 100-325 mesh, to avoid the possible interfering effect of copper, aluminium, magnesium, and other metals upon the test. The fresh and expired *Sodium Diclofenac* (SD) drug marketed under the trade name Divido-75mg (Tabuk /Kingdom Saudi Arabia) was used for corrosion testing. The molecular formula of the drug is $(\text{C}_{14}\text{H}_{11}\text{Cl}_2\text{NO})$ and its chemical structure is shown in Fig. 1. Different amounts of SD were dissolved in acid solution to prepare the desired concentrations in ppm (parts per million). The Evolution™ UV-Visible/300 spectrophotometer (Thermo-Electron), UV-Visible spectrophotometer was used to measure the absorbance of the tested solution. Water bath (Clifton Bata-England). Thermostat (Lauda-Germany).

C. Reagents and Solutions

The stock solution of (5.0 N) Sulfuric acid was prepared from a concentrated acid (98 %, 1.84 g/cm^3 , 98.08 g/mol). The acid was standardized by titration with Standard sodium carbonate (Na_2CO_3). For each

corrosion test, (1.0 N) of sulfuric acid was prepared from the stock solution by diluting with deionized water. The stock solution of 300 ppm of expired *Sodium Diclofenac Drug* was prepared as a stock solution by dissolving 300 mg in deionized water to obtain 1000 ml. Then the concentrations of 30 ppm, 60 ppm, 90 ppm, 150 ppm, 180 ppm were prepared in 50ml flasks. Acetate buffer solution was made up by dissolving 44 grams of sodium acetate in deionized water in a 500 ml volumetric flask. Hydroxylamine hydrochloride 10% solution in water (10 grams dissolved in deionized water in a 100 ml volumetric flask). 1,10-phenanthroline monohydrate. The saturated solution in deionized water (0.5 gram 1,10-phenanthroline dissolved in water in a 500 ml volumetric Pyrex flask). These reagents were added in the order of 0.5 ml hydroxylamine hydrochloride solution, 4 ml acetate buffer solution and 5 ml 1,10-phenanthroline solution to each iron-containing solution in 50ml volumetric flask, which is shaken after each addition, and the volume then completed with water.

D. Standard Reference Curve

Iron Standard Solution (50 ppm Fe): Dissolve 0.0702 g of ferrous ammonium sulfate in water containing 2.5 ml of 1M sulphuric acid and add sufficient water to produce 500.0 ml. Aliquots of 0, 1, 2, 4 and 7 ppm of the standard iron solutions are accurately measured into 50 ml standard flask and the reagents are added as described above and complete the volume with deionized water. In this determination of iron, the spectrophotometer was set 200-900 nm, and the peak of the absorption band is located at 511 nm. The data obtained for the standard curve were plotted (absorbance against the concentration) and represented in "Fig. 2". It will be noted that the five points determined for this curve follow a straight line and thus conform closely to Beer's law.

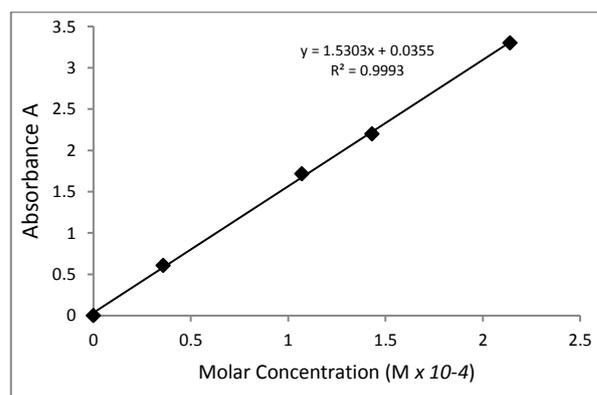


Figure 2: Standard reference curve for iron determination

E. Procedure for Corrosion Test

The pure iron metal was immersed in 1.0 N H₂SO₄ solution with and without different concentrations of an inhibitor of 30 ppm, 60 ppm, 90 ppm, 150 ppm, 180 ppm for desired temperature (303.15K, 323.15K, 343.15K). In each experiment, a constant weight of pure iron powder, 1 g, was added to the tested solution in a 100 ml beaker and placed in a water bath without stirring. The 0.5 ml was withdrawn from tested solution, after 30, 45, 60, 75, 90 min, in standard 50 ml flasks, then the reagent of hydroxylamine hydrochloride, sodium acetate and 1.10-phenanthroline were added. The absorbance was measured in the wavelength range of 200 nm to 800 nm.

F. Procedure for Iron Determination

0.5 ml an aliquot of the tested solution is pipetted into a 50 ml standard flask and the reagents are added, the solution is shaken after each addition. A permanent orange-red complex $[(C_{12}H_8N_2)_3Fe]^{+2}$ develops if any iron is present, the intensity is proportional to the iron content. In this determination of iron, the iron molar concentration is read off directly from the standard reference curve.

III.RESULTS AND DISCUSSION

The UV spectral method was used to estimate the corrosion rate of pure iron in sulfuric acid and its inhibition coefficients and, the kinetics in the presence and absence of *Sodium Diclofenac Drug (SD)*. The UV-VIS spectra were recorded in the range of 200-800 nm for each prepared solution after a specific time. It was observed that the maximum wavelength (λ_{max}) was recorded for *ferrous phenanthroline* complex at 511 nm, this peak was not affected by the addition of the inhibiting drug as in “Fig. 3”.

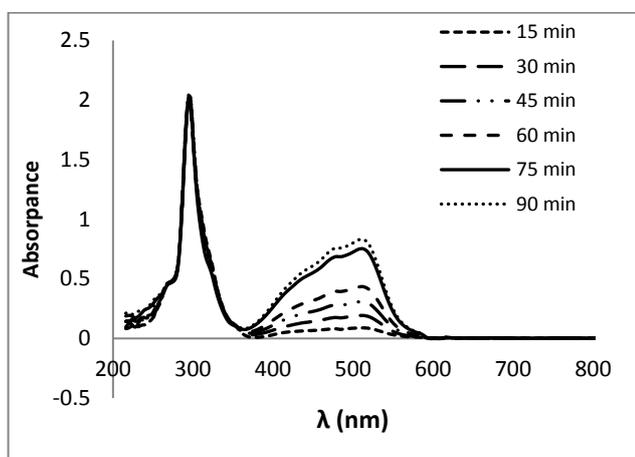


Figure 3: electronic spectrum in the presence of 180 ppm of (SD)

A. Iron Dissolution Rate in Free Sulfuric Acid (R_0)

To obtain a clear picture of the iron corrosion process in a sulfuric acid medium, the corrosion rate of pure iron was studied in the presence of 1.0 N of H_2SO_4 at temperatures of 303.15K, 323.15K & 343.15K. The data obtained in Table 1 were represented graphically in “Fig. 4”.

Table 1: Absorbance of the tested solutions in free H_2SO_4 and the corresponding molar Fe^{2+} concentration at different temperatures

t (min)	Absorption A			Concentration x 10^{-6} (mol/L)		
	303.1 5K	323.1 5K	343.1 5K	303.1 5K	323.1 5K	343.1 5K

30	0.051	0.228	0.866	1.769	9.194	35.96
45	0.075	0.396	1.267	2.776	16.24	52.78
60	0.11	0.619	1.714	4.244	25.59	71.52
75	0.143	0.78	1.813	5.628	32.34	75.68
90	0.178	0.93	1.837	7.097	38.64	76.68

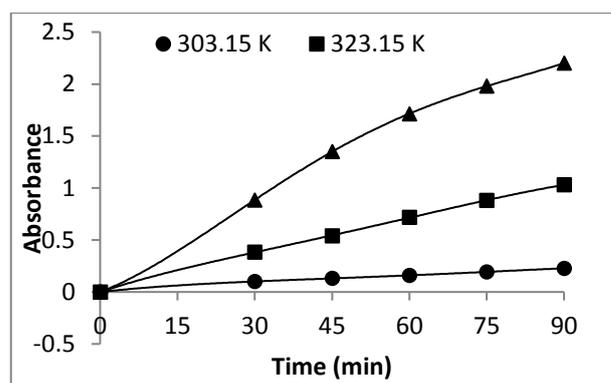


Figure 4: The absorbance vs. time of tested solution in free H_2SO_4 at different temperatures

From “Fig. 4”, we notice that the recorded absorbance increased as the temperature and time increased. Iron dissolution rate (R_0) of pure iron in H_2SO_4 at different temperatures were obtained from drawing the relationship between the concentration of the dissolved iron and time, straight lines were obtained with slope are equal to the reaction rate (R_0) in Molar /min unit, which indicates that the reaction is a zero-order:

$$R = KC^n \tag{1}$$

where $n = 0$. The concentration of the dissolved iron increases with time and temperature as shown in “Fig. 5” and the reaction rate values are shown in Table 2.

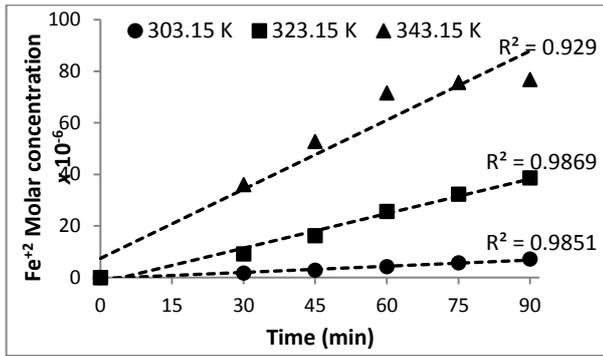


Figure 5: The relationship between time and the dissolved iron concentration in H₂SO₄ at different temperatures

Table 2: Iron dissolution rate in sulfuric acid at different temperatures according to the zero-order reactio

R ₀ X10 ⁻⁶ (M/min)	T (K)
0.0796	303.15
0.4467	323.15
0.8937	343.15

B. Effect of Sodium Diclofenac Inhibitor

Expired Sodium Diclofenac has been tested as an iron corrosion inhibitor using a new chemical-based method of UV-VIS spectroscopy. The absorbance of the visible ray was used to quantify the dissolved iron at different periods and different temperatures. As a result of this method, the reaction rates were determined. Besides, the determination of the inhibition efficiency and the adsorption isotherms were studied to identify the nature of this process.

Table 3: The rate of iron corrosion in sulfuric acid in the presence of different concentrations of the SD inhibitor

SD Conc. (ppm)	R _x 10 ⁻⁶ (M/min)		
	303.15 K	323.15 K	343.15 K
180	0.0668	0.4975	0.899
150	0.0699	0.4821	0.9626
90	0.0744	0.4776	1.092
60	0.0783	0.4363	1.083
30	0.0856	0.4248	1.087

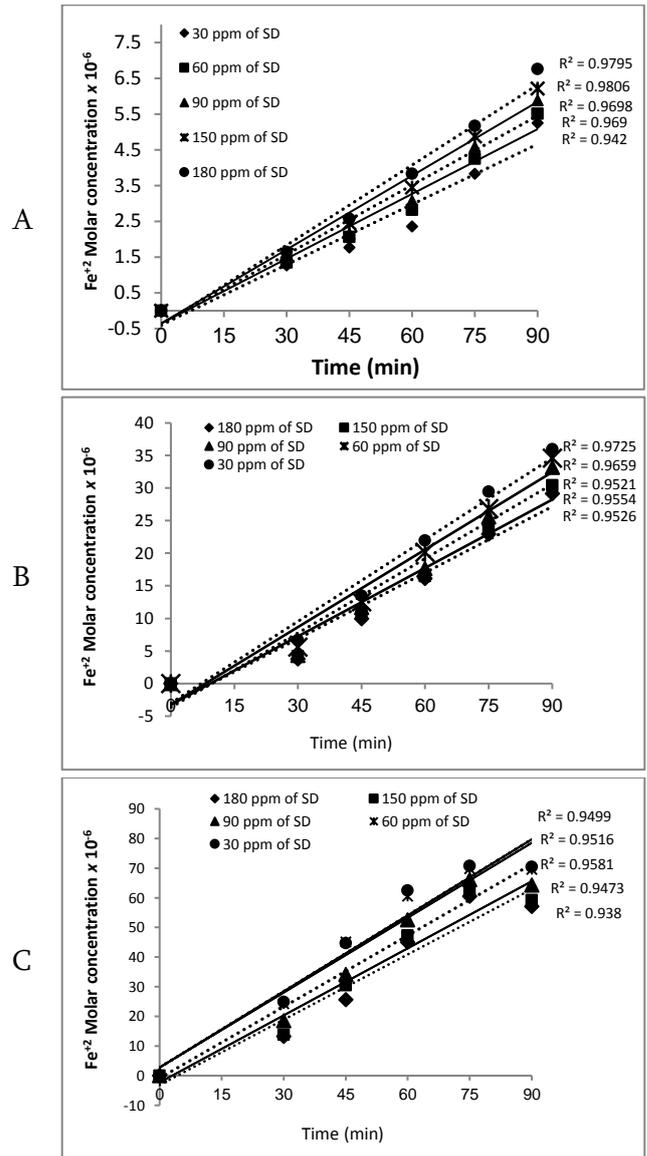


Figure 6: represents the relationship between time and different concentrations of the inhibitor drug at A temperatures at 303.15 K and B at 323.15K and 343.15K

The corrosion rates were determined chemically by measuring the concentration of soluble iron in the acidic medium in the presence of different concentrations of the SD inhibitor. Table 3 shows the effect of the exposure time on increasing the absorption and Fe²⁺ concentration according to the increase in the concentration of ferrous ions over time at different temperatures. Straight lines were obtained by plotting the relationship between Fe²⁺ concentration versus time in the presence of different concentrations of the studied drug as an inhibitor (ppm 180 ppm, 150 ppm & 90 ppm, 60 ppm, 30 ppm)

as shown in “Fig. 6” at different temperatures, which are (303.15K, 323.15K and 343.15K). Through the “Fig. 6”, the reaction rate decreases with increasing SD inhibitor concentration and increases with temperature. The slope of these lines gave the corrosion rate in units of (M⁻¹minutes⁻¹) as presented in Table 3 and “Fig. 7”.

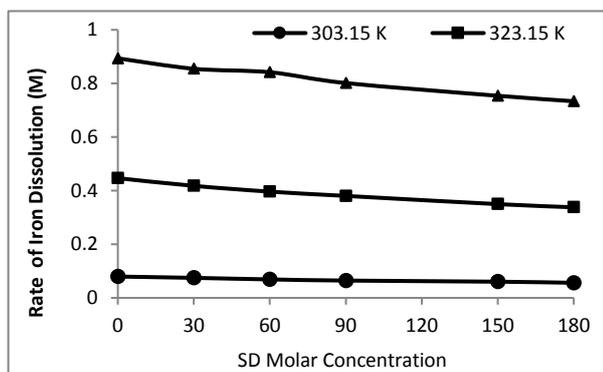


Figure 7: Iron Corrosion rate at different temperatures

C. Inhibition Efficiency

The inhibition efficacy (*Inh*%) of the SD drug was calculated by equation (2) where the absorption in the presence, *A*, and absence, *A*₀, of the SD inhibitor were used in the efficiency calculation.

$$Inh \% = \left(\frac{A_0 - A_{inh}}{A_0} \right) \times 100 \quad (2)$$

Table 4: Values of the inhibition efficiency of pure iron in sulfuric acid and the presence of different concentrations of the inhibitor and at different times and different temperatures

SD Conc. (ppm)	<i>Inh</i> %		
	303.15K	323.15K	343.15K
180	42.98246	31.74757	29.95455
150	38.59649	28.73786	26.81818
90	35.52632	24.27184	23.77273
60	29.82456	19.41748	19.09091
30	25.4386	15.92233	15.45455

The inhibitory behaviour at different temperatures were presented in Table 4 and in “Fig. 8”. This behaviour can be demonstrated based on the

interaction of inhibitor particles with the surface of iron particles that leads to adsorption on its surface physically or chemically. It was noticed that the efficiency increases by increasing inhibitor concentration.

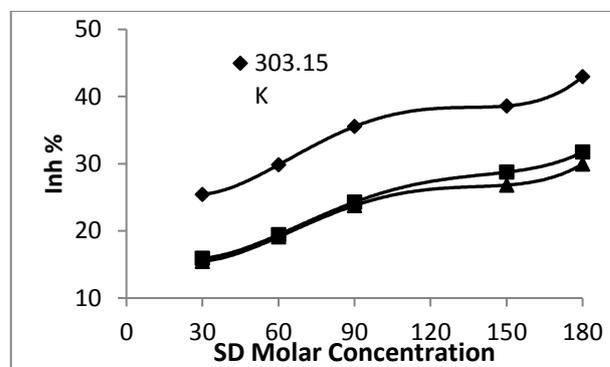


Figure 8: Change of the inhibition efficiency (*Inh*%) with the inhibitor concentration in the acidic medium at different temperatures (A) 303.15 and (B) 323.15 and (C) 343.15 K

D. Effect of Temperature

The effect of temperature on the rate of corrosion of pure iron in sulfuric acid has been studied at different temperatures (303.15, 323.15 and 343.15 K) and different SD concentrations of ppm 180 ppm, 150 ppm, 90 ppm, 60 ppm, and 30 ppm. It is clear that the rate increases with increasing temperatures as shown in Table 3. Thermodynamic functions were calculated at different concentrations of the inhibiting drug as shown in “Fig. 9 & 10” by using of Arrhenius equation and Transition–State equation. Arrhenius equation (equation 3) describes the variation of corrosion rate with temperature.

$$\ln R = \left(-\frac{\Delta E_a^*}{RT} \right) + \ln A \quad (3)$$

Where *R* is the corrosion rate, *T* is the absolute temperature, *A* is Arrhenius pre-exponential frequency factor, *R* is the universal gas constant and ΔE_a^* is a quantitative characteristic of the adsorption process with dimensions of that of energy known as activation energy and which is used to describe the kinetics of the inhibition process. However,

transition–State equation (equation 4) gives the relation between $\ln(R/T)$ vs. $(1/T)$.

$$\ln\left(\frac{R}{T}\right) = \left[\ln \frac{R}{N^{\circ}h} + \frac{\Delta S_a^*}{R} \right] - \frac{\Delta H_a^*}{RT} \quad (4)$$

Where h is Plank constant, N° is Avogadro's number, and both ΔH_a^* and ΔS_a^* are the enthalpy and entropy of activation for the corrosion process, respectively [11]. The enthalpy and entropy of activation are deduced from the slopes and the intercepts of the plot. The slopes and intercept obtained from “Fig. 9 & 10” were used to evaluate activation energy, enthalpy and entropy changes of the system and the values obtained are presented in Table 5.

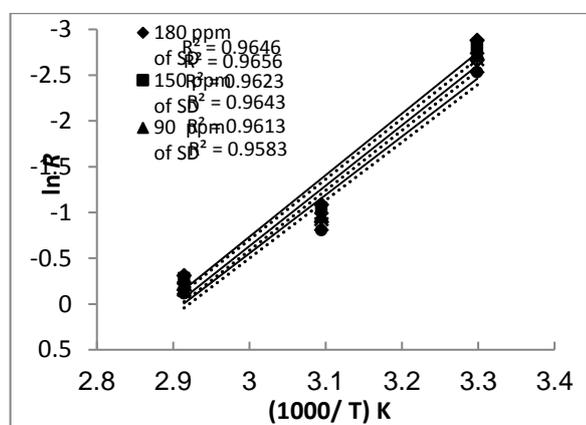


Figure 9: Applying the Arrhenius equation to calculate the activation energy in the absence and presence of the SD inhibitor

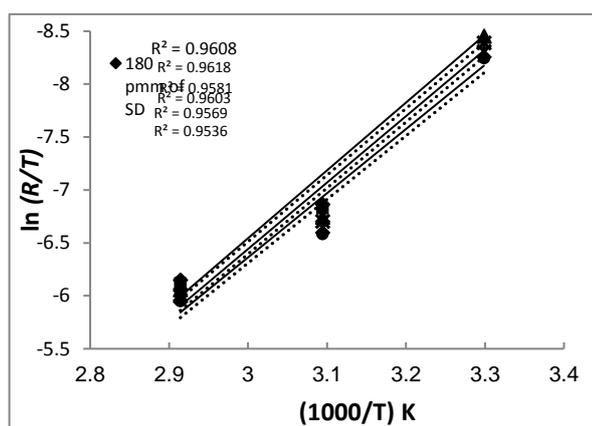


Figure 10: Transition–State plot for the corrosion rate of iron in acidic medium at different temperatures

Table 5: represents thermodynamic functions at different concentrations of inhibitor drug

SD	$+\Delta E_a^*$	$+\Delta H_a^*$	$-\Delta S_a^*$
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Conc. (ppm)	KJmol ⁻¹	KJmol ⁻¹	Jmol ⁻¹
180	55.935	53.214	92.169
150	54.957	52.278	94.829
90	54.84	52.164	94.597
60	54.496	51.818	95.245
30	53.086	50.408	99.21
0	52.685	50.008	99.96

Table 5 gives the activation parameters of temperature influence on the corrosion rate of iron in the absence and presence of SD inhibitor with 1.0 N sulfuric acid. The positive values of ΔE_a^* and ΔH_a^* in the absence and presence of SD inhibitors reflect the endothermic nature of the iron dissolution process [17]. The negative values of ΔS_a^* in the absence and presence of the SD drug are indicated an unspontaneous process.

F. Adsorption Isotherm Curves

To obtain the adsorption isotherm, the degree of surface coverage (θ) for various concentrations of the inhibitor (C) has been calculated. Attempts were made to fit the θ values to Langmuir and Kinetic-thermodynamic model of adsorption isotherm.

Langmuir Adsorption isotherm (equation 5) can be used to calculate the adsorption constant (K_{ads}).

$$\theta / (1-\theta) = K_{ads} C \quad (5)$$

“Fig. 11” representing a Langmuir Adsorption isotherm at different temperatures (303.15 K, 323.15 K & 343.15K), Where the slope represents the equilibrium constant K_{ads} . The equilibrium constant of the adsorption process is related to the standard free energy of adsorption ΔG_{ads} by:

$$\Delta G_{ads}^{\circ} = -R \ln(55.5K_{ads}) \quad (6)$$

where ΔG_{ads}° is the free energy of the adsorption, R is the gas constant and T is the temperature, calculated values of the free energies are also presented in Table 7. The values obtained from this research were 4.38, 6.12 and 6.67 kJ mol⁻¹ at 303.15K, 323.15K, and 343.15K, respectively, which indicates that the adsorption mechanism of SD on the iron particle in H₂SO₄ solution at the studied temperatures is

physisorption [18]. The lower values of K_{ads} for studied SD, Table 7, indicate weak adsorption on the iron particle surface [19, 20].

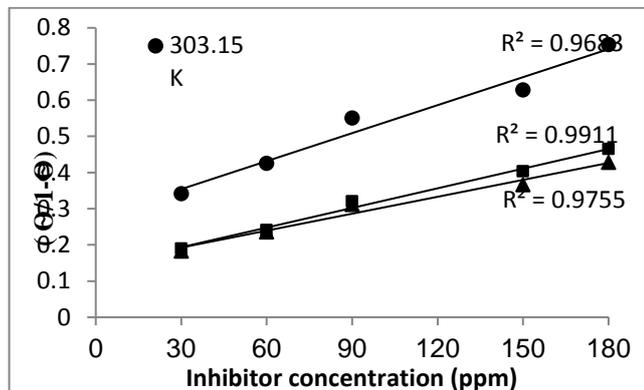


Figure 11: Langmuir adsorption isotherm plot of DS drug

The Kinetic-thermodynamic model of adsorption isotherm [21] (equation 7) was tested for its fit to the experimental data.

$$\log\left(\frac{\theta}{1-\theta}\right) = \log k' + y \log C \quad (7)$$

y is the number of inhibitor molecules occupying one active site and k' constant related to the binding constant of adsorption of the process of adsorption. The plot of $\log\left(\frac{\theta}{1-\theta}\right)$ versus $\log C$ "Fig. 12", would give a straight line slope equal to y , Table 7.

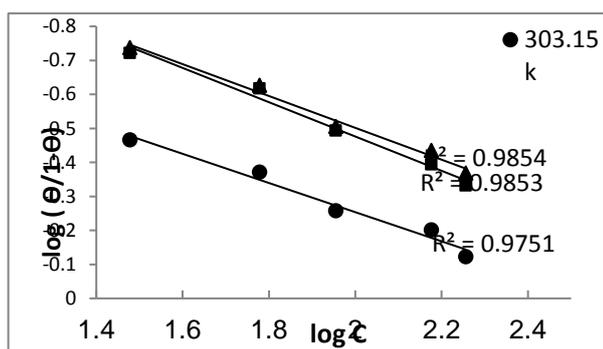


Figure 12: Thermodynamic-kinetic adsorption isotherm plot of DS Absorption

Table 7: Some calculated factors from the application of adsorption curves

T	Y	K_{ads}	$-\Delta G_{ads}KJ/mol$
303.15	0.429	2.6×10^{-3}	4.38
323.15	0.504	1.8×10^{-3}	6.12
343.15	0.468	1.6×10^{-3}	6.67

It's obvious in Table 7 that, the values of y obtained are less than the unit and indicate that the molecules drug involved in the adsorption process covers two of the active site on the metal surface.

G. Theoretical Calculations

The optimized molecular structures with minimum energies obtained from the DFT calculations are given in "Fig. 13".

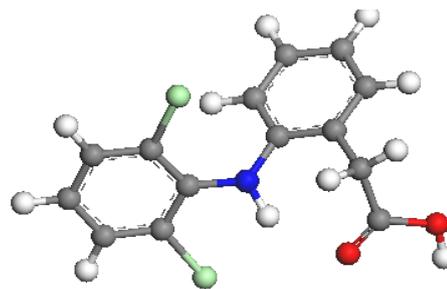


Figure 13: The computer-optimized drug portion, which contains 8 single-level backs that contain the entire portion

Data obtained from quantum chemical calculations using DFT shows LUMO, HOMO distributed along with inhibitor molecules. However, the oxygen (O) atom in the inhibitor molecules is only LUMO contributed. The highest occupied molecular orbital (HOMO), and the lowest unoccupied molecular orbital (LUMO) were used to predict the adsorption active site of the SD inhibitor molecule. According to the frontier molecular orbital theory, the formation of a transition state of chemical inhibitor is due to an interaction between HOMO and LUMO of the reacting inhibitor[22-26]. The energy of HOMO describes the electron-donating ability of a molecule and characterizes the tendency of the molecule towards attack by electrophiles. A high value of E_{HOMO} (- 4.695) indicates a tendency of the molecule

to donate electrons to acceptor molecules with low energy molecular orbital or empty electron orbital [22, 25]. The LUMO energy characterizes the electron-accepting ability and the susceptibility of the molecule towards attack by nucleophiles. A low value of E_{LUMO} (-1.36 eV), indicates an electron-accepting ability of an inhibitor molecule [22] as shown in "Fig. 14".

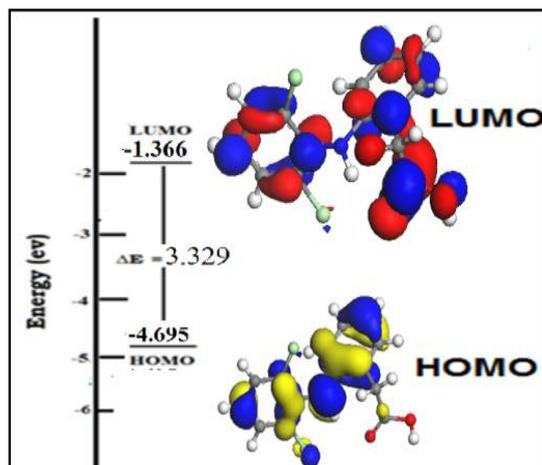


Figure 14: A computer-optimized inhibitor molecule that constructs HOMO & LUMO orbits

The gap energy, $\Delta E_{(LUMO-HOMO)}$, is another essential factor that defines the reactivity of inhibitor molecules towards adsorption on metal surfaces[27]. The large value of the energy gap implies poor inhibition efficiency [28].

The inhibition by *Sodium Diclofenac* can be explained based on physical adsorption as shown in Fig 15". An interaction occurs between the inhibitor particles and the surface of the iron according to:

- The electrostatic force between a positive charge in the NH_2^+ group with sulfate ions adsorbed on the iron surface.
- The attraction between the free electron pair of the π electrons in the aromatic ring and N & O atoms with the vacant orbitals on Fe atoms.

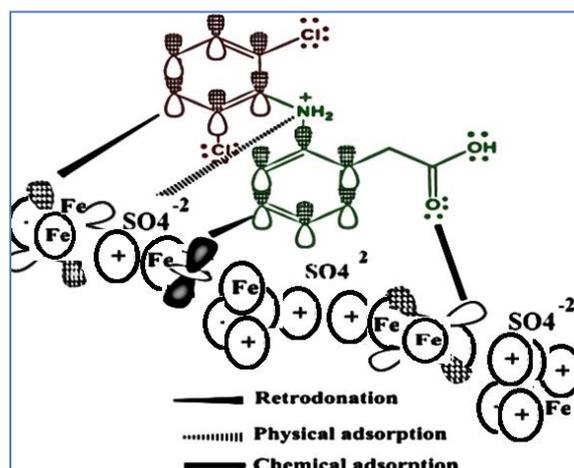


Figure 15: Adsorption mechanism of SD drug on iron particles

IV.CONCLUSION

The inhibitory behaviour of *Sodium Diclofenac* on the corrosion of iron in H_2SO_4 was studied by the UV-VIS spectroscopy technique. The main conclusions can be summarized as follows:

- Sodium Diclofenac* can be used as a corrosion inhibitor in a narrow range where the highest efficiency (42%) was obtained when 180 ppm was used.
- The inhibition efficiency increases with increasing inhibitor concentration and decreasing temperature.
- The activation energy values and the heat content indicate that the process is endothermic.
- The adsorption on the iron surface in sulfuric acid was obeyed the Langmuir and Kinetic-thermodynamic model of adsorption isotherm which confirms the physical adsorption.
- Adsorption process indicates that each active site is occupied by two molecules of inhibiting drug.
- according to the DFT calculations, HOMO & LUMO, the drug is adsorbed by heterogeneous atoms (O & N) and the π -electrons in phenyl rings by physical interactions.

V. ACKNOWLEDGMENT

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VI. CONFLICT OF INTERESTS

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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