Wihania somnifera extract as a friendly corrosion inhibitor of carbon steel in HCl solution

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Abstract

There are many methods used to control the dissolution of metals. The use of inhibitors is one of such processes. Once chromates and some organic compounds were used as corrosion inhibitors. However, environmental scientists do not encourage this because chromates and those organic derivatives are toxic in nature. So, researchers are looking for environmentally friendly corrosion inhibitors such as extracts of plant materials. Several plant materials have been used for this purpose. Wihania somnifera (WSE) was studied as a friendly corrosion inhibitor against carbon steel corrosion in 1 M hydrochloric acid by mass reduction (MR), potentiodynamic polarization (PP), electrochemical frequency modulation (EFM) and electrochemical impedance spectroscopy (EIS) techniques. Surface analysis was performed on carbon steel with and without Wihania somnifera using an atomic force microscope (AFM). The impact of temperature was studied and explained. Investigation of experimental data denote that the inhibition efficiency (IE%) raises with raising concentration of Wihania somnifera extract while decreases with raising temperature. The activation and adsorption thermodynamic parameters such as ΔS^* , ΔH^* and E_a^* were calculated and explained. The negative sign of ΔG^0 indicates that the process is spontaneous. Potentiodynamic polarization data identified that this Wihania somnifera extract acts as a mixed inhibitor and is physically adsorbed on carbon steel surface following the Langmuir adsorption isotherm. The charge transfer resistance increases, while the double layer capacitance decreases with an increase in Wihania somnifera extract concentration. The data from various techniques are in good agreement.

Keywords: corrosion inhibition, Wihania somnifera extract, carbon steel, HCl, EIS, mass reduction, potentiodynamic polarization.

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Introduction

Carbon steels (CS) have wide application in industry and in our everyday lives due to their amazing properties, and moreover, it is the foremost ubiquity sort of steel alloys utilized in many industrial communities. Carbon steel is considered the foundation for the larger part of business and finds worldwide utilization in tremendous amounts. Every year petrochemical plants, oil and gas generation utilize colossal amounts of CS [1, 2]. One disadvantage of carbon steel is that it is readily damaged in humidity and corrosive media.

In order to remove rust and other corrosion products, pickling operations are performed utilizing strong acids [3-6]. Corrosion mitigation of CS in direct contact or completely immersed in a corrosive medium can be done effectively by many corrosion inhibitors. One of the methods to protect metals and alloys from corrosion is by the use of inhibitors. Numerous researches demonstrated that increasing inhibitor concentrations and decreasing the concentration of an acid reduce the undesired metal disintegration, *i.e.*, control the damaging impact of corrosion [7-12].

The aim of this paper is the use of environmentally friendly materials as inhibitors for CS corrosion in 1 M HCl solution, and the choice was to use *Wihania somnifera* to protect CS from corrosion. The use of plant extracts was chosen due to their cheapness and availability. In this research, several methods were utilized to examine the protective behavior, including electrochemical and chemical studies, as well as analysis of the metal surface by different techniques.

Experimental

Materials and solutions

Table 1 shows chemical composition of the utilized CS.

Table 1. Chemical composition (weight %) of CS specimens.

Element	C	Mn	P	S	Si	Fe
%wt.	0.2000	0.3500	0.0390	0.040	0.360	99.0110

Preparation of plant extract (Wihania somnifera)

Wihania somnifera were gathered, dried in an oven at 70°C for 6 hours, then crushed. 50 grams of the Wihania somnifera powder was refluxed in 500 ml bidistilled water for one hour. The aqueous solution was filtered and concentrated to 100 ml. This solution was utilized to prepare solutions with various concentrations by dilution.

Chemical measurement (Mass reduction, MR)

Carbon steel specimens with identical geometrical dimensions ($2 \times 2 \times 0.2$ cm) were weighed before dipping in 100 ml of HCl solutions in the presence and absence of various concentrations of *Wihania somnifera* extract. All the corrosive environments were exposed to air. The tests were performed in 1 M HCl to compare with a sample that was placed in a solution of 1 M hydrochloric acid with various concentrations of *Wihania somnifera* extract. Samples were left for half an hour in solutions, then collected and dried. The samples are weighed and then placed again in the same solutions. The steps were repeated every half an hour until the end of the experiment after 2.5 hours [13]. The k_{corr} (corrosion rate) of the CS sample were computed utilizing the following relationship:

$$k_{\rm corr} = \frac{\Delta W}{At} \tag{1}$$

where ΔW is the mass reduction, A is the sample surface area exposed to the solution in cm², and t in min is the immersion time. The average MR was measured and IE % and the degree of surface coverage (θ) were calculated from the equation:

$$IE\% = 100 \cdot \theta = \left[1 - \frac{W}{W^0}\right] \cdot 100 \tag{2}$$

where W^0 and W are the MR data of CS in 1 M HCl (blank) and in solutions inhibited by Wihania somnifera extract, correspondingly.

Electrochemical techniques

Electrochemical tests were performed in a cell involving three classic electrodes, one of which was the CS working electrode that was studied. Prior to each experiment, the surface of CS coins was mechanically polished with altering grades of emery papers and rinsed by bidistilled water. The exposed surface area was 1 cm². The second electrode was the reference electrode and the third electrode was the auxiliary electrode [14]. The working electrode was placed for 30 minutes in the solution used at the open circuit potential, where a stable state was reached.

Electrochemical frequency modulation (EFM) measurements

Two frequencies, namely 2 and 5 Hz, were used for potential perturbation, while a frequency of 0.1 Hz was used in the EFM test. The CF-2 and CF-3 causality factors, I_{corr} , β_a and β_c were calculated by using the largest peeks [15, 16].

Electrochemical impedance (EIS) measurements

The open circuit potential (OCP) was used and current signals were measured in frequency range from 100 kHz to 10 Hz for impedance measurements [17]. The θ and IE% were determined by the following Equation 3:

$$IE\% = 100 \cdot \left[1 - \frac{R_{\text{ct(free)}}}{R_{\text{ct(inh)}}} \right]$$
 (3)

Where $R_{\text{ct(free)}}$ and $R_{\text{ct(inh)}}$ are the resistances with and without Wihania somnifera extract, respectively.

Potentiodynamic polarization (PP) tests

The PP scan was used to record Tafel curves by sweeping the working potential from -700 to 700 mV at a scan rate of 0.5 mV s⁻¹. I_{corr} was calculated by extrapolation of cathodic and

anodic Tafel lines to give $\log I_{\text{corr}}$ and E_{corr} . θ and IE% were derived from the EIS results using the Equation 4 below.

$$E\% = 100 \cdot \left[1 - \frac{I_{\text{corr(inh)}}}{I_{\text{corr(free)}}} \right] = \theta \cdot 100$$
 (4)

where $I_{\text{corr(inh)}}$ and $I_{\text{corr(free)}}$ are the corrosion currents in the presence and absence of *Wihania* somnifera extract, correspondingly.

Surface Analysis

Atomic force microscopy (AFM) test

This technique provides a 3d dimensional picture of the CS corroded surface and gives information about the corrosion process. This can be done by using a Pico SPP2100 AFM device that provides the roughness of the surface that can be evidence of the inhibition of *Wihania somnifera* on a CS surface.

Fourier transform infrared (FTIR) test

Thermo Fisher Scientific, Waltham, MA, USA was used to record FT-IR spectra that give us information about the inhibitor structure based on peaks corresponding to the functional groups of chemical compounds in the *Wihania somnifera* inhibitor.

Results and Discussion

Mass reduction (MR) test

Data on corrosion rate, θ and *IE*% of carbon steel in 1 M HCl solution with various concentrations of *Wihania somnifera* extract are given in Table 2. The MR of carbon steel in the acid environment with and without various concentrations of *Wihania somnifera* extract after various immersion times (30–150 min) at 25°C are given in Figure 1. The *IE*% improved with an increase in the concentration of the *Wihania somnifera* extract. The inhibition is due to the adsorption of *Wihania somnifera* extract on the carbon steel and formation of a layer from adsorbed *Wihania somnifera* molecules on carbon steel surface which separates the carbon steel surface from the acid solution and blocks the corrosion sites [18–20].

Adsorption isotherms

Wihania somnifera extract is adsorbed on the CS surface because the energy of interaction between the CS surface and the extract is larger than that between H₂O molecules and the metal [21]. The Langmuir adsorption isotherm fits the data obtained perfectly (Figure 2). The Langmuir adsorption isotherm is expressed as:

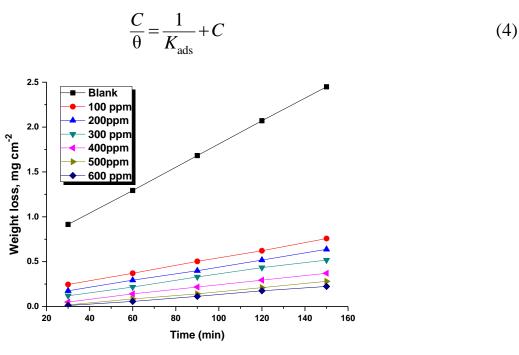


Figure 1. MR-time plots for CS corrosion with and without various concentrations of *Wihania somnifera* extract.

Table 2. Influence of various concentrations of *Wihania somnifera* extract on k_{corr} (mg×cm⁻²×min⁻¹) and *IE*% of CS in 1 M HCl at various temperatures.

Conc. ppm	25	°C	30	°C	35	°C	409	PC	459	°C
	k_{corr}	IE%	k_{corr}	IE%	k_{corr}	IE%	k_{corr}	IE%	$k_{ m corr}$	IE%
Blank	0.116	_	0.014	_	0.192	_	0.419	_	0.513	_
100	0.034	70.7	0.047	66.5	0.068	64.3	0.1609	61.6	0.2098	59.1
200	0.023	79.9	0.045	68	0.064	66.6	0.1496	64.3	0.1975	61.5
300	0.018	84.1	0.038	72.7	0.055	71.3	0.1307	68.8	0.172	66.3
400	0.014	87.6	0.030	78.5	0.046	76	0.1110	73.5	0.1488	71
500	0.012	89.5	0.025	82.2	0.037	80.4	0.088	78.8	0.1185	76.9
600	0.010	91.3	0.020	85.3	0.031	83.6	0.0758	81.9	0.1067	79.2

The plot of C vs. C/θ of Wihania somnifera at various temperatures is shown in Figure 2. This plot gives straight lines with an intercept equals to $1/K_{ads}$. The change in the free energy of adsorption (ΔG_{ads}^0) was determined from the following relation:

$$\Delta G_{\text{ads}}^0 = -RT \ln \left(55.5 K_{\text{ads}} \right) \tag{5}$$

The $\Delta G_{\rm ads}^0$ values at all temperatures are reported in Table 3. The changes in adsorption enthalpy ($\Delta H_{\rm ads}^0$) were determined from the following equation:

$$\log K_{\text{ads}} = \left(\frac{-\Delta H_{\text{ads}}^0}{2.303RT}\right) + constant \tag{6}$$

The plot of log $K_{\rm ads}$ vs. 1/T is shown in Figure 3. The slope of the line is $(-\Delta H_{\rm ads}^0/2.303 \text{R})$, the $\Delta H_{\rm ads}^0$ data were reported in Table 3. Then the following equation (7):

$$\Delta G_{\rm ads}^0 = \Delta H_{\rm ads}^0 - T \Delta S_{\rm ads}^0 \tag{7}$$

was used to obtain $\Delta S_{\rm ads}^0$ at various temperatures. Table 3 shows the adsorption parameters obtained for the *Wihania somnifera* extract. The negative $\Delta G_{\rm ads}^0$ values obtained confirm the spontaneous adsorption of *Wihania somnifera* on the CS surface. The values of $\Delta G_{\rm ads}^0$ smaller than $-20~{\rm kJ/mol}$ indicate that physical adsorption is observed [22]. The $\Delta H_{\rm ads}^0$ value is negative, which means that adsorption of *Wihania somnifera* is exothermic. The fact that the $\Delta H_{\rm ads}^0$ value is less than 40 kJ/mol refers to a physisorption process. The positive $\Delta S_{\rm ads}^0$ value obtained is due to an increase in disorder due to desorption of water molecules from the CS surface [23, 24].

Table 3. Langmuir adsorption parameters of *Wihania somnifera* extract on the CS surface at various temperatures.

Temp., K	$ extbf{\emph{K}}_{ m ads}, extbf{M}^{-1}$	$-\Delta G_{ m ads}^0, { m kJ \cdot mol^{-1}}$	$-\Delta H_{\rm ads}^0$, kJ·mol ⁻¹	$-\Delta S_{\rm ads}^0,$ $\mathbf{J} \cdot \mathbf{mol}^{-1} \cdot \mathbf{K}^{-1}$
298	67	20.5		67.1
303	62	20.1		66.4
308	59	19.5	28.3	63.4
313	52	19.3		62.6
318	49	18.2		60.3

Thermodynamic parameters

The activation energy (E_a^*) for dissolution of the CS was determined from the slope of log k_{corr} versus 1/T drawn by applying Arrhenius equation (7) (Figure 4):

$$k_{\rm corr} = A \cdot \exp\left(-\frac{E_{\rm a}^*}{RT}\right) \tag{8}$$

Where A is the Arrhenius pre-exponential factor. E_a^* increases in the presence of Wihania somnifera extract.

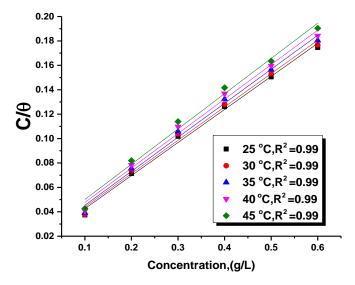


Figure 2. Langmuir plots of *Wihania somnifera* extract on CS surface in 1 M HCl at various temperatures.

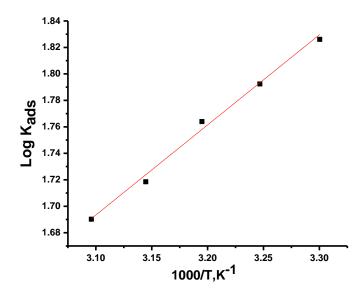


Figure 3. $(\log(K_{ads}))$ vs. (1/T) for the dissolution of CS in 1 M HCl at various temperatures.

The results shown in Table 4 indicate that the activation energy increases with an increase in *Wihania somnifera* concentration due to an increase in the thickness of the barrier layer formed on the CS surface. This increase is due to the adsorption of *Wihania somnifera* extract on the CS surface and corresponds to physical adsorption [25]. The transitional state equation relates the changes in entropy and enthalpy as follows:

$$k_{\text{corr}} = \left(\frac{RT}{Nh}\right) \cdot \exp\left(\frac{\Delta S^*}{R}\right) \cdot \exp\left(\frac{-\Delta H^*}{RT}\right) \tag{9}$$

Where h is the Planck constant. Figure 5, shows straight lines resulting from the plot of log (k_{corr}/T) against 1000/T, and this figure shows the transitional state of the *Wihania somnifera*. The slopes of the curves were utilized to calculate the enthalpy $(-\Delta H^*/2.303R)$, and ΔS^* was obtained from the intercept of the lines. ΔS^* data are negative in the presence of *Wihania somnifera*. These negative data demonstrate that the activated complex in the rate determining step favors coagulation rather than separate molecules in solution, so the lowering in disorder occurred throughout the sequence from the reactant to the starting complex [26, 27].

Table 4. Thermodynamic parameters for dissolution of CS with and without various concentrations of *Wihania somnifera* extract.

Conc., ppm	$E_{ m a}^*$, kJ \cdot mol $^{-1}$	ΔH^* , kJ·mol ⁻¹	$-\Delta S^*, \mathbf{J} \cdot \mathbf{mol}^{-1} \cdot \mathbf{K}^{-1}$
0	64.9	62.2	88.3
100	74.1	71.4	85.3
200	75.3	72.6	65.1
300	75.4	72.7	64.0
400	76.7	74.0	62.7
500	77.3	74.6	62.0
600	79.5	76.8	61.7

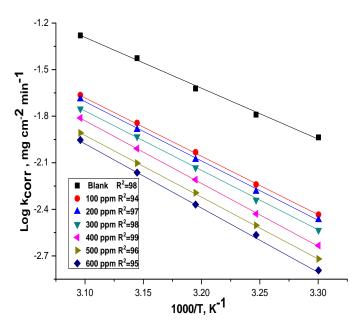


Figure 4. Log k_{corr} vs. 1/T curves for CS corrosion with and without various concentrations of *Wihania somnifera* extract.

PP measurements

PP plots for CS with various concentrations of *Wihania somnifera* extract at 25°C are shown in Figure 6. From the Figure, one can note that both cathodic (hydrogen discharge) and anodic reactions (carbon steel dissolution) were inhibited by addition of various concentrations of *Wihania somnifera* extract. Both β_a and β_c changed in the positive and negative directions, respectively. The electrochemical parameters E_{corr} , β_a and β_c , θ , IE% and i_{corr} were measured and given in Table 5. Data showed that on addition of the extract, the i_{corr} values decreased while the values of E_{corr} (max. shift 18 mV), β_a , and β_c did not change significantly. This indicates that *Wihania somnifera* extract acts as a mixed kind inhibitor [28]. The Tafel lines are almost parallel, which indicates that there is no change in the cathodic hydrogen evolution mechanism and metal dissolution mechanism.

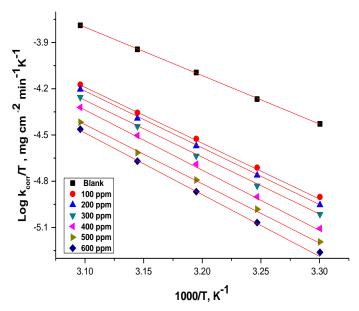


Figure 5. Log (k_{corr}/T) vs. 1/T plots for CS corrosion with and without various concentrations of *Wihania somnifera* extract.

Table 5. Electrochemical parameters, i_{corr} , E_{corr} , β_a , β_c , k_{corr} , θ and IE% of CS corrosion with and without various concentrations of *Wihania somnifera* extract at 25°C

Conc. (ppm)	i _{corr.} μA·cm ⁻²	-E _{corr.} mV (SCE)	$\begin{array}{c} \beta_a \\ mV \; dec^{-1} \end{array}$	$-eta_{c} \ mV \ dec^{-1}$	C. R. mpy	θ	IE%
0	2530	461	124	168	138	_	_
100	1016	462	115	152	11.9	0.598	59.8
200	808	466	121	159	10.5	0.681	68.1
300	693	471	122	142	5.6	0.726	72.6
400	568	476	114	141	4.9	0.775	77.5
500	448	479	115	139	4.4	0.823	82.3
600	372	483	118	148	3.8	0.853	85.3

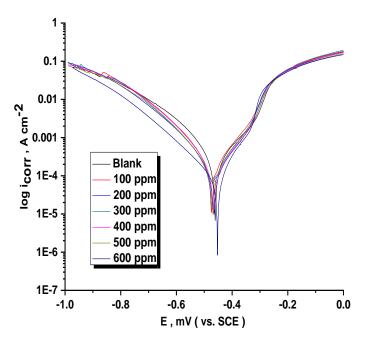


Figure 5. PP diagram for CS corrosion with and without various concentrations of *Wihania somnifera* extract at 25°C.

EFM Tests

The data obtained from EFM for CS in 1 M HCl at various concentrations of *Wihania* somnifera extract at 25°C were measured and are listed in Table 6. The results indicate that i_{corr} decreases with an increase in the concentration of *Wihania somnifera* extract while *IE*% increases. This indicates that this extract is an inhibitor of CS in acid medium. The CF are similar to their theoretical values (2&3). This indicates the validity of the results obtained. The values of i_{corr} can be obtained directly from EFM measurements and independent of the knowledge of Tafel constants. The *IE*% can be calculated as in Equation 3.

Table 6. EFM technique for CS corrosion in the presence and absence of various concentrations of *Wihania somnifera* extract at 25°C.

Conc. (ppm)	i _{corr} μA cm ⁻²	$\begin{array}{c} \beta_a \\ mV \; dec^{-1} \end{array}$	$\begin{array}{c} \beta_c \\ mV \; dec^{-1} \end{array}$	CF-2	CF-3	k _{corr} mpy	θ	IE%
Blank	4763	100	120	2.2	3.0	118	_	_
100	1253	96	110	2.0	3.3	79	0.737	73.7
200	972	90	118	1.9	3.0	73	0.796	79.6
300	867	92	109	2.0	3.2	68	0.818	81.8
400	710	90	110	1.9	2.9	53	0.851	85.1
500	576	91	115	2.2	3.2	49	0.879	87.9
600	472	88	118	1.9	2.8	32	0.901	90.1

EIS measurements

The EIS of CS at the various concentrations of *Wihania somnifera* extract are shown in Figure 7a–b where Figure 7a shows the Nyquist plots and Figure 7b, the Bode plots. The curves show that the each semicircle of the Nyquist plots gradually increases with an increase in the concentration of *Wihania somnifera* extract. The EIS parameters obtained, R_{ct} , the double layer capacitance C_{dl} , and IE% are listed in Table 7. These data showed that there is an increase in R_{ct} and a decrease in C_{dl} . From all the above, a high resistance is established due to adsorption of the *Wihania somnifera* extract at the CS–solution interface. The equivalent circuit that fits the EIS data is presented in Figure 8. The results showed a decrease in the C_{dl} values with an increase in the concentration of *Wihania somnifera*, and this can be explained by a decrease in the local dielectric constant and/or an increase in the thickness of the double electrical layer. This is due to the adsorption of *Wihania somnifera* molecules on the CS/solution interface.

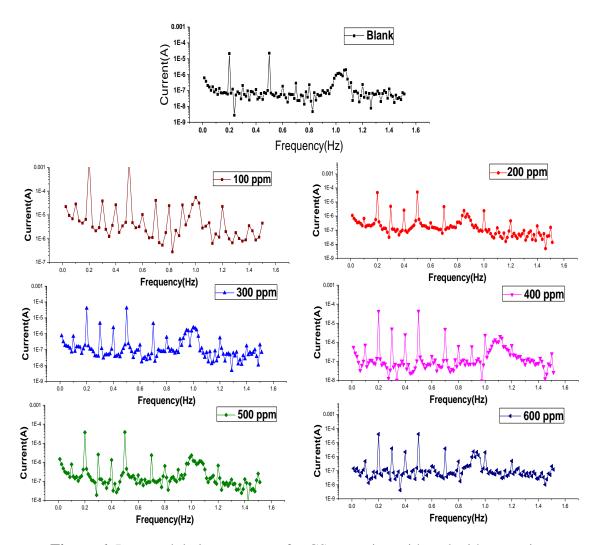


Figure 6. Intermodulation spectrum for CS corrosion with and without various concentrations of *Wihania somnifera* extract at 25°C.

Table 7. Data from EIS measurements	for CS	dissolution	with	and	without	various	concentrations	of
Wihania somnifera extract at 25°C.								

Conc., ppm	$R_{ m ct}, \ \Omega \ { m cm}^2$	C _{dl} , μF cm ⁻²	θ	IE%
Blank	25	385	_	_
100	98	150	0.745	74.5
200	125	113	0.800	80
300	140	108	0.821	82.1
400	162	96	0.846	84.6
500	210	90	0.881	88.1
600	261	80	0.904	90.4

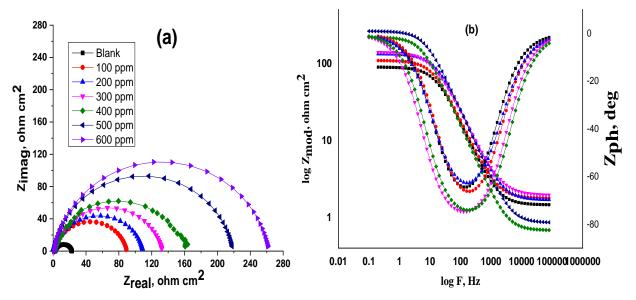


Figure 7. (a) Nyquist and (b) Bode diagrams for CS corrosion with and without various concentrations of *Wihania somnifera* at 25°C.

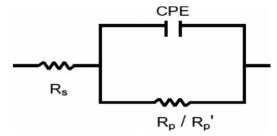


Figure 8. Equivalent model circuit recommended for fitting the EIS data.

AFM analysis

The morphology of CS surface was analyzed using AFM experiments after immersion in 1 M HCl in the absence and the presence of 600 ppm of *Wihania somnifera* extract for 24 h, which affected the surface roughness. The mean roughness profile (Sa) values play an important role in identifying and reporting the efficiency of the inhibitor under study [29]. The roughness plays a role in the explanation of the nature of the adsorbed film on the surface. Figure 9a shows the clear CS surface which displays low roughness (Sa=21), Figure 9b gives the surface of the metal damaged by HCl (blank) which displays a high roughness (Sa=612), and Figure 9c shows the CS surface exposed in the presence of 600 ppm of *Wihania somnifera* extract which was not affected by corrosion and acquired the smallest roughness (Sa=173).

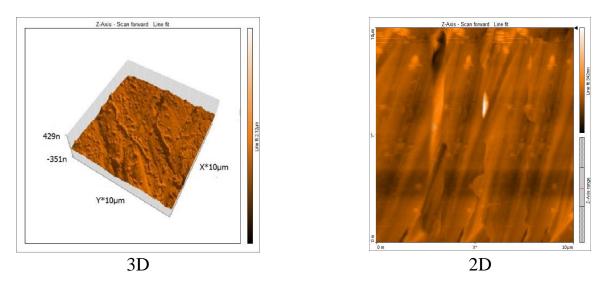


Figure 9a. (3D), (2D) AFM micrograph of CS before immersion in the acid.

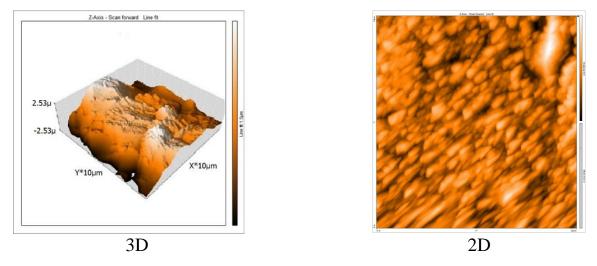
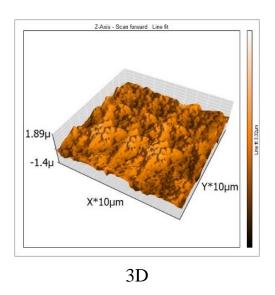


Figure 9b. (3D), (2D) corroding surface of CS after immersion in the corrosive solution at 25°C.



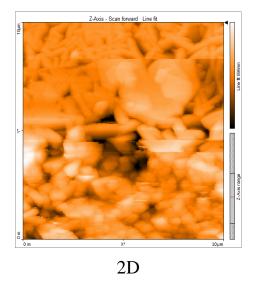


Figure 9c. (3D), (2D) of inhibited carbon steel surface after immersion in the acid solution with 600 ppm of *Wihania somnifera* for 24 hours at 25°C.

Fourier Transform Infrared Spectra (FTIR) analysis

FTIR is a powerful investigative tool to identify the functional groups in a molecule [32]. The FTIR spectrum of *Wihania somnifera* extract is revealed in Figure 10 to show a broad band at 3330 cm⁻¹ due to the stretching vibrations of the N–H bond. The band at 1636 cm⁻¹ corresponds to the vibration of C=N, and that at 1157 cm⁻¹ related to ν (C–O) stretching bands under 1000 cm⁻¹ corresponds to aliphatic and aromatic (C–H) groups. It is observed that there is a shift in the frequencies of the N–H band from 3330 cm⁻¹ to 3387 cm⁻¹. This band that corresponds to the stretching vibrations of C=O shifted from 1636 cm⁻¹ to 1650 cm⁻¹ while the band at 1057 cm⁻¹ related to ν (C–O) stretching vibrations shifted to 1174 cm⁻¹.

Explanation for corrosion inhibition mechanism

This plant mainly contains anthraquinones, flavonoids, polyphenols, lipids, and sterols [30]. Since $E_{\rm corr}$ in the presence of the extract increases compared to the value in its absence and the values of $\Delta G_{\rm ads}^0$ are about 20 kJ mol⁻¹, it follows that the extract molecules are adsorbed physically. In the acid medium the steel surface bears a positive charge, so the protonated extract molecules cannot approach the CS surface to adsorb on it due to electrostatic repulsion. The Cl⁻ ions adsorbed on CS surface generate an extra negative charge near the solution and favor stronger adsorption of cations [31]. Owing to the lone electron pairs, the O and N atoms in *Wihania somnifera* molecules or the protonated molecules may bind with freshly produced Fe²⁺ ions on the CS surface to form CS-inhibitor complexes. These complexes are adsorbed onto the CS by Van der Waals forces to form a protective cover preventing carbon steel from corrosion.

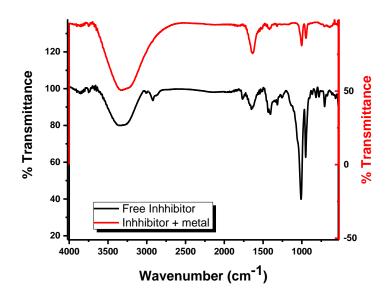


Figure 10. FTIR spectrum of *Wihania somnifera* before and after adsorption on the surface of CS at 25°C.

Conclusions

The inhibition of the dissolution of CS in 1.0 M hydrochloric solution by Wihania somnifera was determined by MR, PP, EIS and EFM measurements. Wihania somnifera can be used as an effective corrosion inhibitor for CS in 1 M HCl solution. The adsorption of Wihania somnifera was found to obey the Langmuir adsorption isotherm. The IE% increases with an increase in the concentration of Wihania somnifera extract and decreases upon increase in the temperature of the medium. Wihania somnifera extract was adsorbed physically on CS surface. The PP data showed that Wihania somnifera extract acts as a mixed type inhibitor. The results obtained from electrochemical and non-electrochemical techniques are in excellent agreement.

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