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# **Corrosion Inhibition and Adsorption Behavior of Nerium Oleander Extract on Carbon Steel in Hydrochloric Acid Solution**

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Nerium Oleander (N. Oleander) *was* investigated as a corrosion inhibitor for carbon steel (CS) in 1 M HCl solution using electrochemical techniques (potentiodynamic polarization (PP), electrochemical impedance spectroscopy (EIS) and electrochemical frequency modulation (EFM)) and nonelectrochemical method (weight loss (WL)). The data showed that the inhibition protection (%IP) raised and corrosion rate (CR) lowered with raising both NOE concentration and temperature to reach 91% at300 ppm. The adsorption of the NOE on CS surface was found to obey the Langmuir's adsorption isotherm. The PP curves showed that this extract behaved as a mixed type inhibitor. Fourier transform infrared (FT-IR) was utilized to detect the functional groups adsorbed on CS surface. Surface morphology was examined using scanning electron microscope (SEM) *and atomic force microscopy* (*AFM*) *techniques*.

Keywords: Inhibition protection, Carbon steel, N. Oleander extract, HCl, SEM, AFM

# **1. INTRODUCTION**

Alkaline scales in cooling water and boiler pipelines are great problem in industrial processes like a power plant, it reduces the plant efficiency and production yield [1-2].HCl is widely used in removal of scales acid cleaning and industry acid pickling [3-5]. This process is controlled by many inhibitors because of HCl has a corrosive effect to CS. Many organic materials have been studied as corrosion inhibitors due to the existence of active center as polar functions with S, O or N and $\pi$ -electrons bonds atoms in the molecule which have a lone pair of electrons in p-orbital overlapping with vacant dorbital of the metal, [6–9], large size of organic inhibitors covers a larger area so it blocks a lot of active centers. Recently, eco-friendly substances are now widely used as corrosion inhibitors due to its nontoxic, biodegradable readily available and economic considerations (must be cheap, easy handling and renewable). Plant extracts have been favored than organic compounds because most of natural products are biodegradable, nontoxic and readily available. Numerous authors have described the usage of the natural products as the potential corrosion protection for different metals and alloys under various conditions [10-19]. N. Oleander is applied as a corrosion protection for CS in HCl solution.

#### 2. MATERIALS AND METHODS

## 2.1 Carbon Steel (CS) Samples

CS used in the study from EL-Atf power plant 750 M.Wt, middle delta for electricity production company, Mahmoudia-El-Behira, Egypt. The chemical structure of CS (wt%) is as next; C (0.17), Mn (0.34), P (0.026), Si (0.003) and Fe (balance).

#### 2.2 Solution Preparation

2.2.1 Preparation of 1M (hydrochloric acid) by diluting the suitable volume of 37 % HCl with bidistilled water.

#### 2.2.2 Preparation of Plant Extract

The dried plant was ground to small pieces and mixing 200 g of the dried plant in 500 ml of water at 80 <sup>o</sup>C for 45 min. The extract N. Oleander was left about 12 hours, then filtered among a glass filter paper then completed by bi-distilled water to 1 liter

#### 2.3Weight Loss (WL) Tests

The CS coins were polished with successive different emery papers (up to 2000 size), rinsed in bi-distilled water and acetone, and weighed by an analytical balance (0.0001 g) [20].

## 2.4. Electrochemical Tests

Three different electrodes cell was used. CS specimens as working electrode (1cm<sup>2</sup>), a reference electrode (saturated calomel electrode (SCE)) and auxiliary electrode (Pt wire). All electrochemical tests were performed utilizing Gamry Instrument (PCI 4-G750) Potentiostat/Galvanostat/ZRA.

## 2.4.1 Potentiodynamic Polarization (PP) Tests

Tafel curves attained from potential (-500 to 500 mV vs SCE) with a scan rate of 1 mVs<sup>-1</sup>

## 2.4.2 Electrochemical Impedance Spectroscopy (EIS) Tests

In order to calculate the impedance of the corrosion process EIS tests carried out by Ac signals, in frequency range from 100 kHz to 0.5 Hz with amplitude of 10 mV peak-to-peak, the corresponding electrical circuit was utilized to clarify the results from impedance.

#### 2.4.3 Electrochemical Frequency Modulation (EFM) Tests

By utilizing two frequencies 2 and 5 Hz with base frequency 0.1 HZ so we obtain a wave repeated after 1 s. The intermodulation spectra contain current responses used to measure the corrosion current density ( $i_{corr}$ ), the Tafel slopes ( $\beta_c$  and  $\beta_a$ ) and the causality factors.

#### 2.5 Fourier Transform Infrared (FT-IR) Analysis

IR Affinity (Perkin-Elmer) spectrophotometer was utilized for recording the FT-IR spectra of the CS and N. Oleander at a central laboratory Mansoura University. A range of frequency = 4500 - 500 cm<sup>-1</sup> was applied to examine the composition of the product from corrosion on the CS surface and the main functional groups in the inhibitor.

#### 2.6 Surface Morphology

The coins utilized for surface morphology investigation were polished, well then dipped in 1 M HCl in the nonexistence (blank) and existence of 300 ppm of aqueous N. Oleander *a*t 25 °C for 24 hours

## 2.6.1 Scanning Electron Microscopy (SEM) Tests

The surface morphology of polished, nonexistence and existence coins was examined by SEM (VEGA3 TESCAN model) were recorded.

## 2.6.2 Atomic Force Microscope (AFM) Tests

The AFM analysis of polished, coins with and without extract was recorded utilizing Nano Surf Easy scan 2 Flex AFM instrument.



Compound d: (Kaempferol)

Scheme 1.chemical structures of different constituents of N. Oleander (daflah)

## **3. RESULTS AND DISCUSSION**

#### 3.1 Weight Loss (WL) Tests

Results of the WL of CS with time for the CS corrosion in corrosive solution containing several concentrations of N. Oleander at 45  $^{0}$ C were recorded in Table 1. From the results, it is obvious that the WL of CS lowered with the raise in the concentration of N. Oleander. This indicates that N. Oleander is adsorbed on CS surface. Table 1 shows the data of (k<sub>corr</sub>) and %IP of N. Oleander at various concentrations and temperatures. The %IP and ( $\Theta$ ) were measured utilizing Eq. (1):

 $\% IP = \Theta x \ 100 = [1 - (CR_{inh}/CR_{free})] \ x100$  (1) where CR<sub>inh</sub> and CR<sub>free</sub> are rates of corrosion with and without extract, correspondingly. The active chemical constituent of  $\pi$ -bonds and oxygen atoms present in the molecules raise the % IP to reach 91.4% at 300 ppm of N. Oleander concentration at 65 °C, also % IP rises with improving the N. Oleander concentrations [21] and temperatures.

Temp., <sup>0</sup> C	Conc., (mg/L)	WL, mg / $cm^2$	CR(k <sub>corr</sub> ) (mg cm <sup>-2</sup> min <sup>-2</sup> ) x10 <sup>-2</sup>	%IP
	blank	8.87	7.39	
	50	3.64	3.03	58.9
	100	3.07	2.55	65.4
25	150	2.67	2.23	69.8
	200	2.38	1.98	73.1
-	250	2.19	1.82	75.3
	300	2.01	1.67	77.3
	blank	11.62	9.69	
	50	4.30	3.58	63.0
-	100	3.65	3.04	68.5
35	150	3.07	2.56	73.5
-	200	2.68	2.24	76.8
-	250	2.39	1.99	79.3
-	300	2.17	1.81	81.2
	blank	17.75	14.79	
-	50	5.28	4.40	70.2
-	100	4.37	3.64	75.3
45	150	3.52	2.93	80.1
	200	3.11	2.59	82.4
	250	2.84	2.36	83.9
-	300	2.43	2.02	86.2
	blank	26.17	21.80	
	50	6.160	5.13	76.4
	100	4.97	4.14	81.0
55	150	3.92	3.27	84.9
	200	3.46	2.88	86.7
	250	3.06	2.55	88.3
	300	2.67	2.23	89.7
	blank	35.49	29.58	
	50	8.23	6.85	76.8
	100	6.31	5.25	82.2
65	150	4.81	4.01	86.4
	200	3.89	3.24	89.0
	250	3.52	2.93	90.0
	300	3.01	2.51	91.4

Table 1. Variations of CR  $(k_{corr})$  and %IP for different concentrations of the N. Oleander at all temperatures used

## 3.2. Influence of Temperature

The deviation in % IP and  $k_{corr}$  with N. Oleander concentrations at altered temperatures were recorded in Table (1). These data exposed that % IP decreases with rise in the N. Oleander concentrations. This can be explained as the extract N. Oleander species are adsorbed on the solution/CS interface where forming a protected film on the CS surface which reduces the corrosion action. Chemical adsorption

mechanism obtained as the data of %IP was raised by improves in temperature [22-23]. The effect of  $k_{corr}$  on the temperature can be stated by next Arrhenius Eq.2:

 $k_{corr} = A \exp(-E_a^*/RT)(2)$ 

where A is the pre-exponential factor. Arrhenius curves obtained for the corrosion of CS in 1 M HCl solution Fig. (1), so we can determine  $E_a^*$  values from the slopes of these linear plots and are listed in Table (2).



Figure 1. Arrhenius plots for CS after 120 min of immersion corrosive solution without and with of various concentrations of N. Oleander

The results showed a higher  $E_a^*$  values in inhibited solutions than uninhibited solutions, this was shown as CS corrode slowly due to the formation of extract film on the CS surface which acts as an energy barrier for the CS corrosion and hence reduce the  $k_{corr}$  [24]. ( $\Delta H^*$ ,  $\Delta S^*$ ) can be calculated from the corrosion process as shown in Eq. 3

 $k_{corr} = (RT/Nh) \exp (\Delta S^*/R) \exp (-\Delta H^*/RT)(3)$ 

A plot of log ( $k_{corr}/T$ ) versus 1/T for CS in 1 M HCl with different concentrations of N. Oleander gives straight lines as shown in Fig. 2. The positive sign of  $\Delta H^*$  designates that the corrosion process is an endothermic one while the  $\Delta S^*$  value is greater and negative suggests that the activated complex prefer an association rather than a dissociation step, so the disorder was reduced [24].

Conc.	Ea	ΔH	-ΔS
ppm	kJ mol <sup>-1</sup>	kJ mol <sup>-1</sup>	kJ mol <sup>-1</sup> K <sup>-1</sup>
blank	30.1	11.8	175
50	16.6	6.1	227
100	14.6	5.1	235
150	11.8	3.9	245
200	10.3	3.3	251
250	10.1	3.1	253
300	8.5	2.5	259

Table 2. Parameters obtained from a	activation of CS corrosion	n without and with of vario	ous concentrations
of N. Oleander in corrosive	solution		



Figure 2. Transition state for CS with and without various concentrations of N.Oleander

#### 3.3. Adsorption Isotherms

The mechanism of  $k_{corr}$  of organo-electrochemical reactions can be explained by using adsorption isotherm. The most commonly utilized isotherms are Langmuir (Fig. 3). Thermodynamic parameters for the adsorption of extract on CS surface in 0.1 M HCl at altered temperatures are recorded in Table (3). The results of Table 3 show negative data of  $\Delta G^0_{ads}$  indicate that the adsorption of N. Oleander on CS in 1 M HCl solution is a spontaneous process [25] also the data are around -20 kJ mol<sup>-1</sup> giving an evidence to the physical mechanism [26].The negative sign of  $\Delta H^0_{ads}$ exposes the exothermic process of the adsorption of extract molecules that leads to either chemisorption or physisorption while endothermic is qualified to chemisorption[27],  $\Delta H^0_{ads}$  data around 100 kJmol<sup>-1</sup> or higher are qualified to chemisorption. This mechanism takes place when the unshared electron pairs in examined molecule interact with dorbitals of Fe to provide protective chemisorbed film [28] .The values of  $\Delta S^0_{ads}$  in the existence of N. Oleander are great and negative that goes together with an exothermic adsorption process [29].



Figure 5. Langmuir adsorption isotherm of N. Oleander on CS in 1M HCl at various temperatures

Table 3. Adsorption parameters of N. Oleander on CS in 1 M HCl at various temperatures

Temp	K <sub>ads</sub>	$-\Delta G^{\circ}_{ads}$	$-\Delta H^{\circ}_{ads}$	$-\Delta S^{\circ}_{ads}$
(°C)	(M <sup>-1</sup> )	(kJ mol <sup>-1</sup> )	(kJ mol <sup>-1</sup> )	(J mol <sup>-1</sup> K <sup>-1</sup> )
25	33	18.9		116.8
35	36	19.7		115.6
45	50	21.3	16	116.7
55	69	22.8		117.8
65	63	20.2		115.6



Figure 4. Log  $K_{ads}$  versus 1/T for the corrosion of CS in 1 M HCl with and without various concentrations of N. Oleander

## 3.4. Electrochemical measurements

## 3.4.1 Open-Circuit Potential (Eoc) Tests

The variation in  $E_{OC}$  with time obtained for CS in 1 M HCl in both with and without N. Oleander at 25°C is plotted in Fig. 5.



Figure 5.  $E_{OCP}$  versus time relatives for CS in 1 M HCl without and with various concentration of N. oleander at  $25^{0}C$ 

The data revealed that  $E_{OCP}$  in the blank solution started at -535 mV and then shifted anodically and after14 min the steady state is obtained, this indicates the initial dissolution process of the air-formed oxide film on the CS surface. In the existence of N. Oleander, the  $E_{OC}$  began at potential relatively negative paralleled with that in the nonexistence of the N. Oleander extract and then shifted anodically. The steady state is attained rapidly, likened with the blank conferring to Riggs [30], from Fig. 7the change in  $E_{OCP}$  on addition of N. Oleander is about 37 mV, revealing that the existence of N. Oleander act as mixed type inhibitor.

#### 3.4.2. Potentiodynamic Polarization (PP) Tests

The outcome data of the influence N. Oleander on polarization curves of CS in 1 M HCl plotted in Fig.6. It is clear that both the cathodic reactions and anodic were inhibited with the appending of N. Oleander, as the extract retard the hydrogen evolution reaction and reduce anodic dissolution. Table 4 shows electrochemical parameters i.e.,  $(E_{corr})$ ,  $(\beta_a, \beta_c)$  and  $(i_{corr})$  obtained from the extrapolation of the polarization bends. We notice slight change in corrosion potential (27 mV) reducing both anodic and cathodic currents. According to Ferreira [31],this shift indicated the mixed-type nature of the extract by the addition of the N. Oleander to the solution The region between linear part of cathodic and anodic branch of polarization curves becomes wider, [32,33]. The results calculated from Tafel polarization displayed best agreement with those obtained from WL. The %IP was obtained from Eq. 4.

$$\% IP = \Theta x \ 100 = (1 - [i_{corr}/i^{o}_{corr}]) x \ 100$$
(4)

where i<sub>corr</sub> and i<sup>o</sup><sub>corr</sub> are the current in existence and nonexistence of inhibitor, correspondingly.



Figure 6. PP curves for the liquefaction of CS in 1 M HCl with and without various concentrations of N. Oleander

Conc	i <sub>corr</sub>	-E <sub>corr</sub>	βa	βc	CR	θ	IP%
ppm	$\mu A \text{ cm}^{-2}$	mV vs SCE	mV dec-1	mV dec-1	mpy		
Blank	1340	513	123	166	612.3		
50	253	486	88	145	115.7	0.810	81.0
100	199	486	79.5	140	90.75	0.850	85.0
150	167	484	78.9	157	76.13	0.870	87.0
200	149	484	72	121.9	68.0	0.888	88.8
250	147	481	70	125.1	67.3	0.891	89.1
300	142	485	71	120.6	64.82	0.894	89.4

**Table 4.** Effect of concentrations of N. oleander on ( $E_{corr}$ ,  $i_{corr}$ ,  $\beta_c$ ,  $\beta_a$ , CR,  $\Theta$ , %IP) of CS in 1 M HCl

#### 3.4.3 Electrochemical Impedance Spectroscopy (EIS) Tests

EIS tests were utilized to study the mechanism of corrosion. The results of Nyquist and Bode diagrams are demonstrated in Figs. 7 and 8, respectively, and these figures displayed a gradual increamet in the semicircle diameter of the Nyquist diagrams by raising the concentration of N. Oleander inhibitor. So the N. Oleander inhibitor retard the corrosion rate [34-35]. From Fig. 7 we noticed that the deviation from an ideal semicircle as a result of frequency dispersion because the inhomogeneity of the surface. Table 4 gives different parameters of impedance as, (R<sub>ct</sub>), (*C*<sub>dl</sub>), (%IE) and electrolyte resistance (*R*<sub>s</sub>). The data of Table 5 demonstrated that, the *C*<sub>dl</sub> data lowered by raising the concentration of N. Oleander extract, this behavior as a result of molecules adsorbed on the surface of CS, and this demonstrated from

Helmholtz model, where the capacitance of double layer was obtain by the next relation. Fig. 9 reveals to equivalent circuit which used in our study [36-38].



**Figure 7.** The Nyquist diagrams of CS in 1 M HCl in the absence and presence of various concentrations of N. Oleander at 25°C



**Figure 8.** Bode diagrams of CS in 1 M HCl in the absence and presence of various concentrations of N. Oleander at 25°C

Conc	R <sub>ct</sub>	C <sub>dl</sub>	ρ	ID 0/
ppm	$(\Omega \text{ cm}^{-2})$	$(\mu F \text{ cm}^{-2})$	0	IF %
blank	18.13	12		
50	84.36	744	0.785	78.5
100	93.73	877	0.807	80.7
150	107.3	761	0.831	83.1
200	107.6	870	0.832	83.2
250	112.9	881	0.839	83.9
300	120.3	725	0.849	84.9

**Table 5.** EIS parameters for CS corrosion without and with various concentrations of N. Oleander extract at 25°C



Figure 9. Circuit utilized for fitting the data of EIS in 1 M HCl

## 3.4.4. Electrochemical frequency Modulation (EFM) Tests

The EFM spectra for N. Oleander in1 M HCl solution at 25°C were shown in Fig. 10. By raising the studied extract concentration the %IP calculated from Eq. 3 increases. Each spectrum is a current response as a function of frequency. Corrosion parameters,  $(i_{corr})$ , $(\beta_a, \beta_c)$  and (CF-2, CF-3), are recorded in Table 6 as a utility of concentrations of N. Oleander at 25°C. The CF in Table 6 are very close to their theoretical data indicating the validity of the results [39].

Corrosion of CS in HCl was found inhibited in existence of N. Oleander. The CS surface property change by introducing N. Oleander as shown from the several techniques results as the extract form protective layer over CS surface reducing  $k_{corr}$ . The surface of the CS were blocked by the adsorption of the extract molecules via adsorption of the protonated (cationic) form of the extract molecules on the negatively charged CS surface by electrostatic attraction (CS is positively charged and becomes negatively charged after the adsorption of the Cl<sup>-</sup> ions on it by electrostatic attraction)



Figure 10. EFM data for CS in 1 M HCl in the absence and presence of N. Oleander

Table 6. EFM technique for CS in 1 M HCl with and without various concentrations of N. Oleand	ler
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Conc	i <sub>corr</sub>	$\beta_a$	βc	$CE_2$	CE-3	k <sub>corr</sub>	IP
(ppm)	μA cm <sup>-2</sup>	mVdec <sup>-1</sup>	mVdec <sup>-1</sup>	CI-2	CI-5	(mpy)	%
Blank	934.2	212	222	1.2	1.8	426.9	
50	217.0	81	98	1.5	3.9	99.1	76.8
100	194.9	80	95	1.9	3.5	891	79.1
150	161.8	78.	91	1.3	3.3	73.9	82.7
200	155.6	78	92	1.4	3.9	71.1	83.3
250	150.3	71	85	1.5	3.8	68.7	83.9
300	148.7	80	93	1.2	3.1	67.9	84.1

# 3.5. FT-IR analysis

Figures 13signify the IR spectrum of extract and that of the film produced on the CS immersed in 300 ppm of N. Oleander the FT-IR spectrum of extract is given in Fig. 13. The stretching -OHfrequency at 3355 cm<sup>-1</sup>, the -CH frequency at 2957 cm-1, the C=O frequency seems at 1637 cm<sup>-1</sup>, the -C=C frequency seems at (1375-1495 cm<sup>-1</sup>) (multiple bands), and the stretching -CO frequency appears at 1054 cm<sup>-1</sup>. The FT-IR spectrum of the film designed on the CS in 1 M HCl and is displayed in Fig. 11. The -OH stretching frequency depleted due to the reaction between the extract and the metal. The stretching aromatic C=C frequency also shifted.





## 3.6. Scanning Electron Microscope (SEM) tests

Figure 12 shows SEM micrographs for surface of CS prior and after its putting in 1MHCl solution without and with the addition of 300 ppm of N. Oleander. As expected, Fig.12A indicates pure surface is evident, whereas in Fig.12B, without N. Oleander, the CS surface is affected by 1 M HCl solution, while in Fig.12C, with extract, the CS surface is no affected by acid corrosion.



**Figure 12.** (A-B-C) SEM images of CS in 1M HCl A) CS, B) 1 M HCl and extract free immersed for 24 h and c) 300 ppm of N. Oleander immersed for 24 h

#### 3.7. Atomic Force Microscope (AFM) Analysis

AFM is a remarkable technique used for measuring the surface roughness with high resolution. Many details about CS surface morphology can be obtained from AFM measurements which help explaining the corrosion process. The three dimensional AFM images were represented in Figure (13).



**Figure 13.** (a) 3D AFM of polished CS, (b) 3D AFM of CS immersed 1M HCl for 1day, (c) 3D AFM image of CS immersed in 1M HCl+300 ppm of N. Oleander for 1 day

Table 7. AFM parameters of N. Oleander at 25°C
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Sample	Roughness average (Sa), nm
Free	134
Blank	464
N. Oleander	39

The roughness calculated from AFM image is summarized in Table (7). The values showed that the roughness rises with adding HCl due to the corrosion occurs on the CS surface but decreased with adding the N. Oleander [40].

## 4. CONCLUSIONS

Upon up the above results, the next conclusions can be drawn:

N. Oleander was found to be an effective, eco-friendly and low cost extract for the corrosion of CS in 1 M HCl. IP% raised with the raise in N. Oleander extract concentrations in 1 M HCl and with rise in temperature. Adsorption of the N. Oleander extract on the CS surface reduces corrosion process obey the Langmuir isotherm. The free energy  $(-\Delta G^{\circ}_{ads})$  negative values of adsorption indicates strong and impulsive adsorption of the N. Oleander extract on the CS surface. Values of Tafel constants  $\beta_a$  and  $\beta_c$  approve that the N. Oleander extract acts like mixed type inhibitor. Rise in R<sub>ct</sub> values and drop in i<sub>corr</sub> and C<sub>dl</sub> values approve that the N. Oleander extract is adsorbed on the CS surface and inhibition process is followed by monolayer adsorption. Chemical and electrochemical methods give inhibition efficiencies with reasonable good agreement

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