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Hydrogen Production and Electrochemical Investigations of an Environmentally Salicornia Extract as a Green Corrosion Inhibitor in a 1 M HCl Solution

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Abstract

A promising method for producing pure hydrogen energy from the dissolution of carbon steel by green inhibitors. Salicornia green inhibitor used for C-steel protection due to both ecological and economic importance (low cost). An evaluation was conducted on the effectiveness of Salicornia extract as a corrosion inhibitor using Potentiodynamic polarization (PDP), electrochemical impedance spectroscopy (EIS), and Hydrogen evolution ,Salicornia are capable of delaying the corrosion of carbon steel (C-steal) in 1 M HCl by forming an adsorbed protective layer of Salicornia molecules. and decrease the reaction of C-steal with the corrosive media consequently hydrogen evolution decreases. The inhibition efficiency for Salicornia inhibitor reached to 93.8% indicating that Salicornia extract acts as good inhibitor. Salicornia molecules adsorb according to the Langmuir isotherm with regression coefficient (R2 = 0.9997). The anticorrosion performance of Salicornia inhibitor was confirmed by Scanning Electron Microscopy (SEM).

Keywords: *Salicornia extract, Corrosion inhibitor, Hydrogen evolution, EIS.*

1. Introduction

In the last few years, one of the biggest issues that cause huge damage during industrial process which cost governments a lot of money especially petroleum field. [1]. Petroleum industries suffer from many problems especially through acid job which required a good green and biodegradable corrosion inhibitors to overcome these problems. C-steel corrosion is one of the biggest issues that face many countries due to numerous of C-steel applications in different industries. Many studies focused on understanding C-steel corrosion process in different environments and different methods to control this process. Using corrosion inhibitors is an effective method for preventing corrosion of metal surfaces through a protection process via formation of insulation layer over metal surface against the corrosive surrounding[2–4]. Natural extract as Salicornia that grown in Middle East especially in west of Iraq (between Sudia Arabia, Syria and Iraq) is considered a good food for animals such as camels. Natural products such as plant extracts, are widely available and cost-effective. As a result, the twenty-first century has seen a surge in green chemistry research and its application in Salicornia. As public understanding of the health and environmental consequences of industrial products comprising toxins has grown, more restrictive regulations on their usage have been enacted in the United States and Europe [5]. With this in mind, the widespread use of conventional Salicornia is being banned because of their toxicity and pollution effect on the environment. This has stimulated great interest in the development of green corrosion inhibitors (Salicornia) among researchers.The family of salicornioideae comprises about 15 genera and 80 species[5] Almost within increasing salt concentration in the roots and shoots, the content of all micro – and macronutrient decrease in the growth medium[6]. Salicornia extract has a mixture of different compounds enriched with aromatic rings, hetero atoms (N&O) and CH aliphatic chain as shown in **scheme.1** [7] showing compounds from 1 - 10 are Ethyl linolenoate, Sitostanol, Octadecyl (E)-ferulate, Eicosanyl (E)-ferulate, Ethyl (E)-2-hydroxycinnamate, Scopoletin, Triacylglycerol of tetracosanoic acid, (E)-6-{[4-methyl-6-((4-methylpentanoyl)oxy) hex-4-enoyl] oxy} heptyl – 6 -ethylheptanoate (Saliramoester) ,1, 10- [Oxybis (4,1-phenylene)] bis (3-hydroxypropan-1-one (Saliramophenone) and 5-Hydroxy-1-(hydroxymethyl)-1H-pyrrole-3-carbaldehyde (Saliramopyrrole) respectively. Recently, scientists use natural product for many industrial applications such as protection of metals from corrosion. Inhibitors such as natural products is suitable to safeguard metals and alloys from corrosion in various

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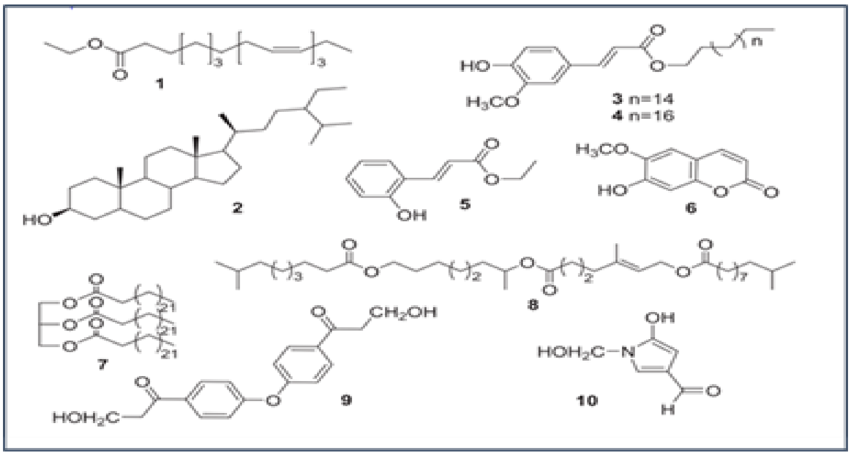
environments [8]. In the present study, Salicornia extract was applied as a corrosion inhibitor for C-steel in acidic solution (1 M HCl). Various techniques were applied for evaluation of Salicornia extract as C-steel corrosion mitigator via employing EIS, PDP, hydrogen evolution and SEM. All these techniques confirmed the adsorption capacity of Salicornia extract on C-steal surface and its inhibition power.



Scheme.1: Salicornia extract.

2. Experimental

2.1. Preparation of Salicornia

Salicornia extracted using ethanol and normal n-hexane at room temperature with stirring continuously for duration of 40 hours. The solution underwent filtration. and the solvent as distilled of using rotary vacuum evaporator.

2.1.1. Preparation of solution

Stock solution of Salicornia extract (1000 ppm) was prepared using the destructive electrolyte (1 M HCl) that was diluted from 37% HCl using distilled water. Different concentrations of Salicornia extract (25 ppm – 250 ppm) were prepared by dilution at room temperature without stirring.

2.1.2. C-steel composition

C-steel in this study composites from C = 0.16%, Si = 0.10%, Mn = 0.40%, S = 0.02%, P = 0.13% and the equilibrium iron. Mechanical polishing of C-steel samples was carried out using various silicon carbide sheets, washed with bi-distilled water then left to dry at room temperature. C-steel working electrode with surface area (1 cm2) was also utilized for all electrochemical analyses.

2.1.3. Hydrogen evolution measurements

C-steel samples with dimensions (3.5 cm × 2.5 cm × 0.5 cm) were immersed in 1 M corrosive HCl solution with and without various concentrations of Salicornia extract and placed into a closed container to prevent any loss of hydrogen gas. The volume of hydrogen gas generated through the corrosion process was practically recorded

2.1.4. Electrochemical technique

A standard three-electrode corrosion cell containing C-steel as a working electrode, Ag/AgCl electrode as a reference electrode, and Pt wire as an auxiliary electrode. At room temperature, C-steel corrosion behavior in 1 M HCl was studied using PDP and EIS techniques in absence and presence of different concentrations of Salicornia extract using Auto lab PGSTAT 128n. PDP with potential range (± 400 mV) against OCP (open circuit potential) and scan rate (1 mV.S-1) was measured. Also, EIS measurements with frequency range (100 kHz - 0.5 Hz) using AC signals at open circuit potential were carried out.

2.2. Scanning electron microscopy (SEM)

Using SEM (Jeol, 5400, Japan) that considers a powerful tool used for C-steel surface examination in absence and presence of high concentration (250 ppm) of Salicornia inhibitor after 6 h of immersion. 2D images of the inhibited and uninhibited sample were carried out at magnification of X = 1000 using energy acceleration beam 30 kV. Also, Fourier-transform infrared spectroscopy (FTIR) was carried out for confirmation about the adsorption of Salicornia molecules over C-steel surface.

3. Results and discussion

3.1. Hydrogen evolution

Fig (1) represents linear link between volume of H_2 production of C-steel with time in the treated and untreated solutions. A notable decrease in hydrogen evolution amount after the addition of Salicornia extract with immersion was observed as in Fig (1) reflecting the adsorption of Salicornia on C-steel surface. From Fig (1), the volume of hydrogen curves in presence of Salicornia doses, fall below the obtained curve of free acid solution expressing that, Salicornia molecules cover C-steel surface forming a protective layer against the destructive particles [9,10]. The inhibition efficiencies (η) of Salicornia inhibitor were determined according to the following equation[11,12]:

$$\eta = (\Delta V_{HCl} - \Delta V_{inh})/\Delta V_{HCl})\ X100 \qquad (1)$$

ΔV_{inh} and ΔV_{Hcl} denote the volume of hydrogen evolution in the presence and absence of Salicornia respectively.

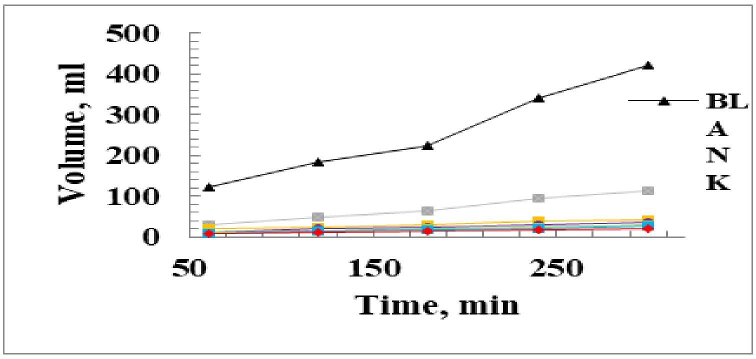


Fig (1): Effect of time on the hydrogen evolution of C-steel in a 1 M HCl solution with and without different Salicornia concentrations.

Data in Table (1) demonstrated that, the mitigation potency of the studied Salicornia increased as their doses increased which can explained by Salicornia adsorption on the surface of C-steel is consequently enhanced C-steel surface coverage which decreased C-steel/HCl contact [13]. The inhibition efficacy of Salicornia increased with its doses and reached 95 % at 250 ppm which can be attributed to Salicornia adsorption via formation of coordination bond between hetero atoms (N, O, and S) and p-electron of C=O group or aromatic rings with vacant d-orbital of iron [14–16]. The film stability and inhibition efficacy of Salicornia with time was investigated via H2 production measurements that showed Salicornia mitigation potency increased with time till reach 95 % at 250 ppm after 5 h. this fact confirmed the adsorption capacity of Salicornia molecules over carbon steel surface and formation of a stable protective film by chemical coordination bond between hetero atoms (N, O, and S) and μ-electrons of C=O group or aromatic rings with vacant d-orbital of iron [15–17].

Table (1): The volume of hydrogen generated and inhibition efficiencies in presence and absence of Salicornia extract with time.

Inh.	60 min		120 min		180 min		220 min		300 min	
	V, ml	η	V, ml	η	V, ml	η	V, ml	η	V, ml	η
Blank	122	----	185	----	225	----	340	----	420	----
25 ppm	31	74.59	48	74.05	63	72.00	94	72.35	113	73.09
50 ppm	22	81.96	25	86.48	30	86.66	38	88.82	42	90.00
100 ppm	13	89.34	20	89.18	24	89.33	30	91.17	36	91.42
150 ppm	11	90.98	16	91.35	21	90.66	25	92.64	30	92.85
200 ppm	9	92.62	14	92.43	18	92.00	22	93.52	26	93.80
250 ppm	7	94.26	11	94.05	14	93.77	19	94.41	21	95.00

3.2. Potentiodynamic polarization technique

Fig (2) demonstrates the salicornia extract's effect on PDP curves of C-steel immerses in 1 M HCl at room temperature. Some corresponding parameters such as (E_{corr}) corrosion potential, (β_a) anodic and cathodic (β_c) slopes and (i_{corr}) corrosion current density were computed and listed in Table (2). The inhibition efficiency (η) was determined using equation:

$$\eta = (i_{corr.HCl} - i_{corr.inh})/i_{corr.HCl}\ X100 \quad (2)$$

where the corrosion current densities in the absence and presence of the Salicornia extract are represented by $i_{corr.HCl}$ and $i_{corr.inh}$ respectively.

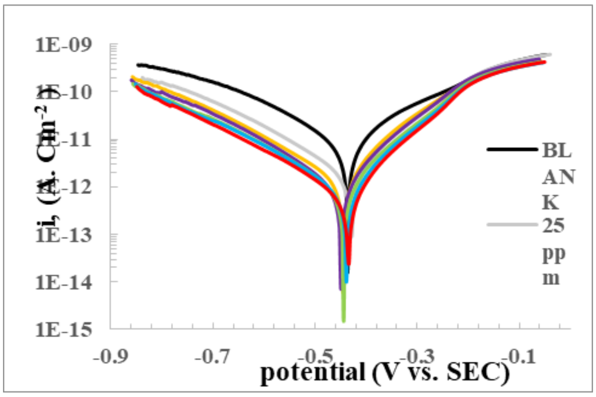


Fig (2): PDP curves of C-steel in 1 M HCl solution with and without different concentrations of Salicornia extract.

Table (2): PDP parameters of C-steel in 1 M HCl solution with and without different concentrations of Salicornia extract.

Inh.	Ecorr, (V)	**β_a (V/dec)**	**β_c (V/dec)**	**i_{corr} (A/cm^2)**	***CR* *(mmy)***	***R_P* *(Ω, cm^2)***	θ	η
Blank	-0.449	0.18462	0.1788	0.000867	10.071	45.513	-----	-------
25 ppm	-0.440	0.12451	0.17276	0.00022	2.5605	142.61	0.745	74.577
50 ppm	-0.458	0.12268	0.17198	0.000168	1.956	184.73	0.806	80.578
100 ppm	-0.452	0.10778	0.16429	0.000116	1.3505	243.20	0.866	86.591
150 ppm	-0.447	0.09675	0.1544	7.98E-05	0.92685	323.86	0.908	90.797
200 ppm	-0.436	0.08780	0.15637	6.85E-05	0.79643	356.30	0.921	92.092
250 ppm	-0.432	0.08238	0.15737	5.37E-05	0.6236	437.59	0.938	93.808

From Table (2), the addition Salicornia shifted the inhibition efficiency to higher values and current density to lower value indicating the adsorption capacity of Salicornia molecules on C-steel surface via their active centers forming a barrier layer on the surface of C-steel which decrease rate of metal dissolution [17,18]. The values of β_c and β_a change slightly and no definite change in E_{corr}, suggesting that the mixed-type inhibitory action of salicornia extract and blocking both cathodic and anodic sites through the same mechanism [18–20]. At low anodic potential the adsorption rate of the studied Salicornia inhibitor increases and creating a covering insoluble layer over metal surface indicating that the inhibition behavior depends on the Salicornia concentrations. while, at higher anodic potential (> -0.226 mV), Salicornia has a larger desorption rate than adsorption rate as the corrosion current density increases as result in dissolution rate of C-steel increases [21,22]. As in Fig (2), the displacement of PDP curves towards the less active area signifies the inhibitory impact of Salicornia extract [23–25]. Also, the same appearance of PDP curves of C-steel in absence and even after addition of different doses of Salicornia extract confirmed that, the corrosion mechanism of C-steel remained unchanged. This observation can be also confirmed by the parallel appearance of cathodic lines [26–28]. The η value of Salicornia extract as in Table (2) increased till reach 93.808 % at 250 ppm indicated its inhibition power and shielding of extra C-steel surface area via construction of an insulation layer that protected C-steel surface from the aggressive attack of HCl solution [29].

3.3. Electrochemical impedance spectroscopy (EIS)

The effectiveness of the Salicornia extract in controlling C-steel corrosion in 1 M HCl solution at 25 ºC was evaluated using EIS technique showing Nyquist and Bode-Phase angle graphs, as depicted in Fig (3). The imperfect shape of Nyquist impedance can be explained by frequency dispersion and surface roughness on C-steel [29]. Nyquist curves displays a single capacitive loop with diameter increases with Salicornia concentrations enhancement [30,31]. The addition of Salicornia extract decreased C-steel corrosion rate by forming of a barrier layer covering it surface which prevented the C-steel from being ionized which can be noticed in augmentation of the diameter of the Nyquist curves as seen in Fig (3).

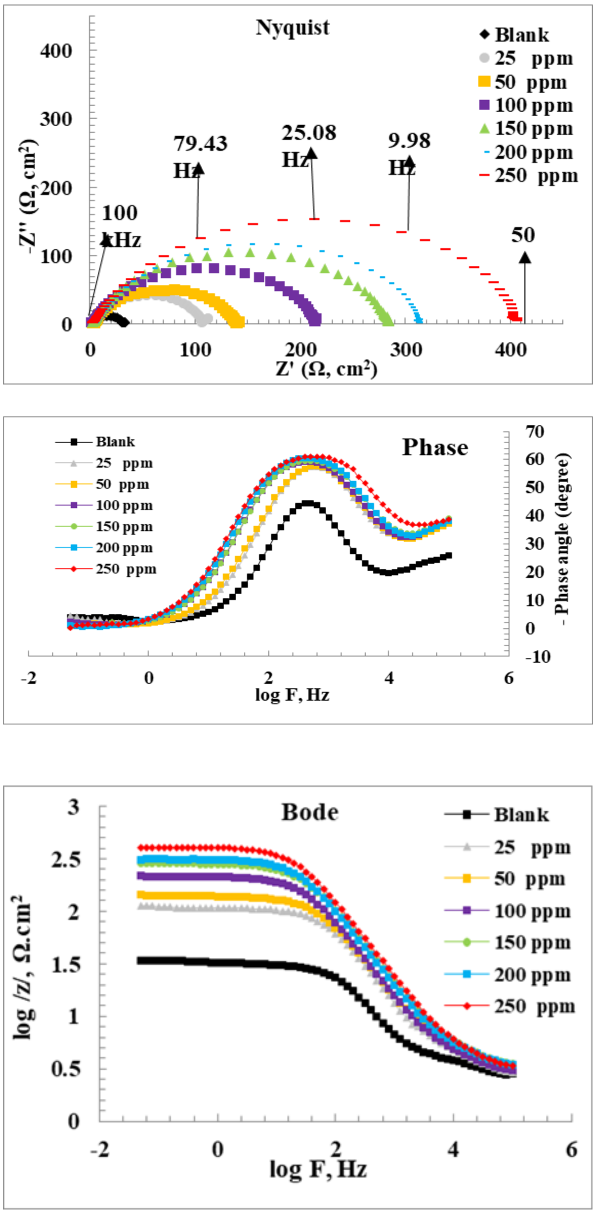


Fig (3): Nyquist, Bode and Phase diagrams of C-steel in 1 M HCl with and without various concentration of Salicornia extract at room temperature.

Also, the addition of Salicornia extract shifted log/Z/ to higher values and phase degree towards -90. This behavior demonstrated the adsorption impact of Salicornia inhibitor [32,33]. Also, Nyquist curves of C-steel before and after addition of Salicornia extract with the same shape verified that, C-steel corrosion mechanism didn't change and controlled by charge transfer [34]. Nyquist and Bode-Phase curves in the absence and presence of Salicornia extract with single time constant showing equivalent circuit of C-steel reaction has been proposed as shown in Fig (4) with solution resistance, denoted as R_s, is accompanied by the constant phase element (CPE), which may be characterized by Y^o and coefficient n which are parameters associated with the heterogeneity of the metal surface and R_{ct} is the charge transfer resistance [35].

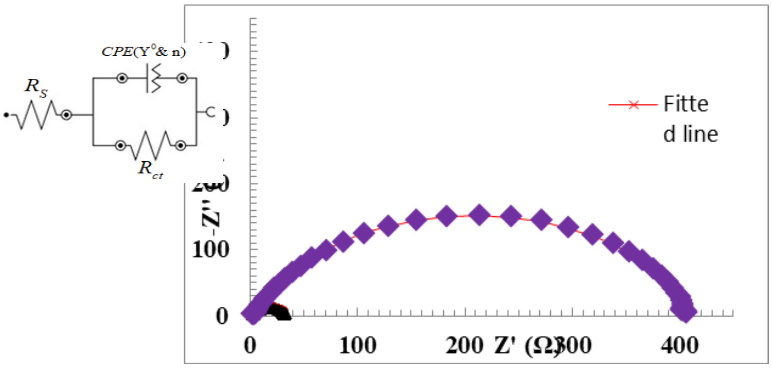


Fig (4): Fitted EIS experimental data using the equivalent circuit for C-steal in the absence and presence of 250 ppm of Salicornia extract.

The increase in n values as in Table (3) indicated that, C-steel surface inhomogeneity decrease due to Salicornia molecules' adsorption [36]. Also, Bode-Phase module shows more capacitive response which means corrosion rate decreases as concentrations of Salicornia increase giving drops in C_{dl} values which can be determined using equation [37]:

$$C_{dl} = 1/(2\pi R_{ct}\, F_{img\rightarrow Max}) \quad (3)$$

Where $F_{img \rightarrow Max}$ is the frequency at the maximum Z_i (imaginary resistance).

Table (3): EIS parameters of C-steel in 1 M HCl with and without various concentrations of Salicornia at room temperature

Inh.	RS (Ω.cm2)	***Rct. (Ω.cm2)***	***CPE*** **Y^O, sn Ω^{-1}** cm^{-2} ×10^{-5}	N	***C_{dl}*** (F/cm^2) ×10^{-5}	***R_P* (Ω, cm^2)**	θ	η
Blank	3.4997	28.20	4.26	0.841	4.49	45.513	-----	-------
25 ppm	3.5448	104.56	2.00	0.849	2.41	142.61	0.730	73.029
50 ppm	3.1385	136.62	1.88	0.851	2.32	184.73	0.793	79.358
100 ppm	3.6586	212.91	1.79	0.855	2.36	243.20	0.867	86.754
150 ppm	3.6301	282.09	1.63	0.854	2.25	323.86	0.900	90.003
200 ppm	4.3665	313.67	1.54	0.857	2.02	356.30	0.910	91.009
250 ppm	5.1415	407.22	1.42	0.859	1.96	437.59	0.930	93.075

Data in Table (3) shows that C_{dl} values decrease with rising of concentrations of Salicornia extract reflected C-steal protection from the corrosion process via replacing corrosive particles with inhibitors molecules which clarifies the role of Salicornia extract in C-steal protection [38–40]. Using the values of R_{ct} in the presence and absence of Salicornia, the values of η were determined as in Table (3) as follow:

$$\eta = (R_{ct.inh} - R_{ct.b})/R_{ct.inh})\, X100 \qquad (4)$$

Where $R_{ct.inh}, R_{ct.blank}$ are the charge transfer resistance of C-steal in presence of Salicornia extract and in free 1 M HCl respectively [41]. As the concentrations of Salicornia increase, the values of R_{ct} increase according to the adsorption of Salicornia molecules and decrease C_{dl} values of by replacement of solution (water) molecules from the surface [42,43]. Salicornia molecules adsorbed on metal surface formed a protective layer which its thickness increases as concentration increase giving consequently increase in inhibition efficiency [44,45]. Salicornia extract demonstrated a high level of effectiveness in controlling C-steel corrosion in 1 M HCl and touch 93.07 % at 250 ppm.

3.4. Adsorption isotherm:

The adsorption of Salicornia extract was studied using θ (surface coverage) values obtained from PDP method. After applying different isotherm models, Langmiur isotherm was the best model for explanation Salicornia adsorption according to the following Formula [46]

$$C/_{\theta} = \left(\frac{1}{K_{ads}}\right) + C \qquad (5)$$

Where K_{ads} is the adsorption binding constant. The straight line in Fig (5) with regression coefficient $R^2 = 0.9997$ and the value of slope obtained from Linear relationship was approximately 1 indicating that, the adsorption isotherm of Salicornia extract follows Langmuir isotherm [47] . ΔG_{ads} (Gibbs free energy) was calculated using K_{ads} value as follow [46].

$$\Delta G^{\circ}_{ads} = -RT\ln(10^6 K_{ads}) \qquad (6)$$

Where R is universal gas constant.

The calculated value of Kads in Table (4) reflected a strong interaction between Salicornia and C-steel surface via electron sharing process (donor-acceptor) forming high stable insulation layer of the adsorbed Salicornia molecules. Also, the value of ΔGads in Table (4) was-28.053 which inferred that, the adsorption of Salicornia molecules on C-steel surface was spontaneously and hybrid adsorption process (mixed physical and chemical adsorption). This demonstrated that, Salicornia extract suppress C-steel corrosion rate in both corrosive media via electrostatic interaction (physical adsorption) between the charged sites of both Salicornia molecules and metal surface besides formation of chemical bond with 3-d orbital of iron (chemical adsorption) via hetero atoms (N, O, and S) and μ-electrons of C=O, C=C or aromatic rings in Salicornia structure [43]. All these observations exhibited the higher adsorption probability of the studied Salicornia inhibitor over C-steel surface and its key role in C-steel corrosion control via formation of a protective defensive layer that decreased the contact between C-steel surface and the corrosive surrounding consequently decrease the corrosion rate.

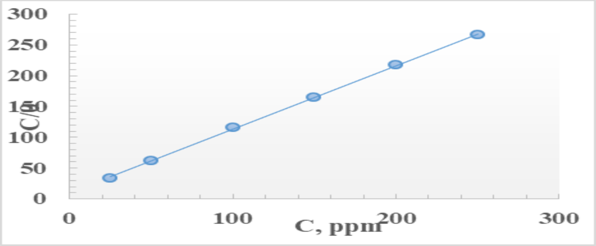


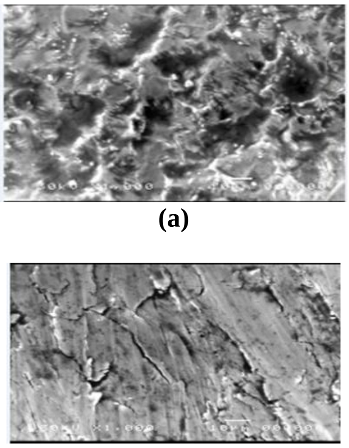
Fig (5): Langmuir isotherm for adsorption of Salicornia extract over C-steel surface in 1M HCl.

Table (4): Langmuir isotherm parameters for Salicornia extract at room temperature.

Inh.	Slope	Intercept	R^2	Kads, $L.mol^{-1}$	kJ mol^{-1}	ΔGads, kJ mol-1
Salicornia	1.0324	9.968	0.9997	0.1003	-28.053	-28.053

3.5. SEM analysis

C-steel surface analysis using SEM which is an effective tool confirming the protection power of the studied Salicornia parallel with the electrochemical measurements. Fig (6) showed the corrosion performance of C-steel in 1 M HCl free and with 250 ppm of Salicornia extract in after immersion time 6 h showing 2D images of C-steel in 1 M HCl (Fig (6a)) with damaged and destroyed surface owing to the aggressive action of HCl (corrosion process) [19,48,49]. On the other hand, the addition of Salicornia extract reflected its anticorrosion behavior as in Fig (6b) showing C-steel with tangibly improved surface compared with that in the uninhibited solution which can be explained by Salicornia protection role in C-steel corrosion through its adsorption process and construction of an insoluble film layer which decreasing the contact between corrosive HCl and C-steel [22,36].


(a)

(b)

Fig (6): SEM for C-steel in 1 M HCl free (a) and containing 250 ppm of Salicornia extract (b).

Also, Fourier-transform infrared spectroscopy (FTIR) was carried out for more data about the adsorption of Salicornia molecules over C-steel surface as shown in Fig (7) with a typical band of O-H for passivate metals at 3406 cm-1 and a notable band at 1635 cm-1 corresponds to C=C of aromatic rings [23,37]. The inhibition efficiency of Salicornia extract was compared with other published works as presented in Table (5).

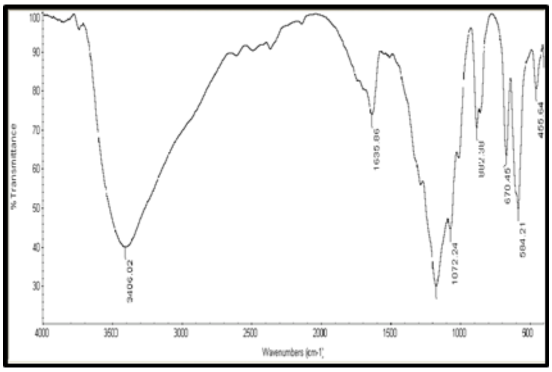


Fig (7): FTIR of salicornia adsorbed on C-steel surface.

Table (5): Comparison between the inhibition efficiency of salicornia extraction with other green inhibitors.

Green inhibitors	***η***	**Reference**
Loquat leaf extract	42.3	[51]
Ilex paraguariensis extract	91.4	[52]
Cynara scolymus extract	89.2	[53]
Green tea extract	87.1	[54]
Tamarind shell tannin	87.7	[55]
Salicornia extract	93.07	Present study

3.6. Salicornia Inhibition mechanism.

Using Salicornia extract, the inhibition effect of C-steel in 1 M HCl was verified by the formation of an insoluble protective film layer over C-steel surface that reduced the contact between the surface and the corrosive acidic solution through the adsorption process of Salicornia inhibitor which can be explained by substitution or replacement of H_2O molecules from C-steel surface with inhibitors molecules by accumulation at metal/solution interface. Also, Salicornia molecules adsorbed physically via electrostatic attraction on the surface of C-steel between charged atoms on its structures and C-steel and chemically by formation of coordination bond between electron pair on heteroatoms or π-electrons of the aromatic/heterocyclic ring with iron orbitals. This interaction causes the corrosion rate of C-steel to decrease, indicating that Salicornia extract acts as an effective inhibitor.

4. Conclusion

1. Green inhibitor such as Salicornia extract was evaluated as corrosion inhibitor for C-steel in 1 M HCl solution.
2. Different electrochemical techniques such as PDP and EIS were carried out.
3. The inhibition efficiency recorded 93.808 % and 93.705 % based on PDP and EIS respectively.
4. Hydrogen evolution rate in presence of Salicornia extract was under control reveals that Salicornia extract acting as effective inhibitor.
5. SEM examination showed that, Salicornia protected C-steel from the corrosive action of HCl solution via the adsorption process on C-steel surface.
6. Langmuir adsorption isotherm with R^2= 0.9977 governed Salicornia molecule's adsorption process over C-steel surface.

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