Inhibition of the pitting corrosion of stainless steel 304 in 0.5 M HCl by clove extract

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Abstract

An environmentally friendly green inhibitor based on clove flower buds aqueous extract was applied for corrosion inhibition of stainless steel 304 surface in 0.5 M HCl solution. Applying integrated experimental techniques (electrochemical combined with weight loss and surface morphological) the clove flower buds aqueous extract inhibition properties were examined. Potentiodynamic polarization curves indicated that clove extract acts as anodic type inhibitor. EIS measurements showed that the charge transfer resistance and consequently the inhibition efficiency increase with increasing concentration of clove with values above 90% at 12 ppm extract. The results obtained from the weight loss method confirmed those of electrochemical techniques. The results proved that the corrosion process was controlled by adsorption of the clove molecules on the stainless steel 304 surface through diffusion control. The optical micrographs are in agreement with those of electrochemical techniques which lead to confirmation of the ability of clove aqueous extract to protect stainless steel 304 and to retard its corrosion in 0.5 M HCl solution. Application of the adsorption isotherms have been evaluated and indicated that the adsorption mechanism is best described by each of the Flory–Huggins isotherm and kinetic–thermodynamic model. However, Langmuir isotherm is not applicable which clarified that the adsorption process is non-ideal.

Keywords: *stainless steel, clove extract, adsorption, acidic, optical microscope, EIS, polarization.*

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I. Introduction

Stainless steels have many applications, and widely used in chemical processing equipment, nuclear stations, heat exchangers, pharmaceutical and medical activities [1–3]. Stainless steels have excellent resistance to corrosion in different atmospheric environments and many corrosion solutions, this resistance to corrosion is mainly due to the existence of some elements such as chromium, nickel and molybdenum in composition of steel and the presence of thin passive layer of chromium oxide (Cr_2O_3) on its surface [4]. Nevertheless,

the corrosion resistance of stainless steel is limited by its susceptibility to pitting corrosion in presence of chloride ions [5, 6]. Several methods have been used to fight against corrosion; however, the most effective method is corrosion inhibition. Organic molecules containing sulphur, nitrogen or oxygen atoms have good inhibition efficiency towards the corrosion of steel in acid media. Several research articles are directed to find non-toxic, environmentally- friendly and cheaper inhibitors [7, 8]. Novel approaches and current trends in the field of inhibition science and technology for corrosion protection of metals and alloys have been discussed in two recent review articles. [9, 10].

In previous works [11–16] the inhibition of corrosion of carbon steel, aluminum, zinc and copper metals in various media have been investigated using different plant extracts. The objective of this paper is to investigate the inhibition of the pitting corrosion of 304 stainless steel in 0.5 M HCl by clove extract. Weight loss method, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) techniques were used to study the corrosion inhibition characteristics of the clove extract and the optical microscope was used to examine the morphology of the surface of stainless steel.

II. Experimental

II.1. Electrochemical tests

Electrochemical impedance and polarization curves were recorded using ACM 604 Instrument. The frequency range for EIS measurements was $0.1 \leq f \leq 1.0 \cdot 10^4$ Hz with applied potential signal amplitude of 10 mV around the rest potential. Polarization were carried out at scan rate 30 mV/min. The data were obtained in a three-electrode mode cell; graphite rod and saturated calomel electrode were used as counter and reference electrodes. The specimens used for constructing the working electrode were stainless steel rods that had the following chemical composition (wt.%): Cr 18.2; Ni 9.85; Mn 0.81; Si 0.59; Cu 0.075; C 0.067; P 0.031; and Fe in balance. The stainless-steel samples were fixed in poly tetrafluoro ethylene (PTFE) rods by an epoxy resin in such a way that only one surface was left uncovered. The exposed area (0.2728 cm^2) was mechanically polished with a series of emery papers of variable grades, the samples were then washed thoroughly with distilled water followed by analytical grade ethanol and finally with distilled water, just before insertion in the cell. Measurements were done at 30°C.

II.2. Weight loss method

Wight loss studies were carried out at 30°C. The rectangular coupons with 5 cm^2 area of stainless steel with the same chemical composition of the electrode sample were used in experiments. They were polished, cleaned and immersed in 100 mL of 0.5 M HCl solution free from or containing different concentrations of the extract. The weight loss studies were carried out for 72 hours. Corrosion rates (weight loss per cm^2 per hour) were calculated.

II.3. Optical microscopic examination

Optical micrographs have been taken by using an Euromex optical microscope with color video camera that is connected to a personal computer. The microscope provides magnifications up to 40x.

II.4. Plant

Clove was used in this study as a natural inhibitor. It is growing in various geological parts of Asia and South America. It has pharmacological properties such as antimicrobial, anti-inflammatory, analgesic, anti-oxidant and anticancer activity, other identified activities such as its anti-ulcerogenic potential and effect on osteoporosis and especially its effect on the central nervous system (CNS) encompassing seizure control, Parkinson's disease, antidepressant effects [17]. The main component in the essential oil extracted from cloves is eugenol, comprising 72–90% [18]. Large quantities of eugenol are used in detergents and soaps for their spicy aroma but they cause discolouration due to their phenol structure. Other constituents include essential oils acetyl-eugenol, beta caryophyllene, and vanillin [19]. The molecular structure of eugenol is represented in Figure 1.

Figure 1. Chemical composition of eugenol.

II.5. Preparation of clove extract

Stock solution of clove flower buds was extracted by refluxing 5 g of the dry material in 250 mL distilled water for one hour. The refluxed solution was filtrated to remove any contamination. The concentration of the stock solution was calculated in $\text{g}\cdot\text{L}^{-1}$ by heating till dryness 5 mL of the stock solution and reweight after drying. The concentration of the stock solution was expressed in terms of ppm.

II.6. Preparation of the test solutions

Double distilled water and analytical reagent-grade HCl (37%) were used for preparing solutions. 12.5 mL of 4 M HCl is added to an appropriate volume of the stock solution of the clove extract and double distilled water in 100 mL flask to obtain solution of 0.5 M HCl and the required concentration of the extract.

III. Results and Discussion

III.1. Potentiodynamic polarization results

Figure 2 shows the polarization curves of stainless steel in aqueous solutions of 0.5 M HCl in absence and presence of different concentrations of clove extract. It is obvious from the figure that, the polarization curves show activation behaviour followed by formation of protective passive film on the steel surface at the different passivation potentials (E_{pp}). During upward scanning, breakdown occurs, indicating starting of the pitting corrosion as a result of the aggressive attack of Cl^- ions at the pitting potential (E_p) in the trans-passive region of the polarization curve, which evident by a sudden increase in the current density.

Figure 2. Polarization curves for stainless steel 304 in 0.5 M HCl solution in absence and presence of different concentrations of clove extract.

Values of the associated electrochemical parameters such as, corrosion potential (E_{corr}), corrosion current density (i_{corr}), cathodic Tafel slope (β_c), percent inhibition of the extract ($\%P$), polarization resistance (R_p), pitting potential (E_p), passivation potential (E_{pp}) and passivation current density (i_{pass}) are collected in Table 1.

In the active region, the data reveals that the addition of the clove extract leads to decreasing markedly the corrosion current density (i_{corr}) and shifting the corrosion potential (E_{corr}) to more noble potentials. This effect is probably attributed to the adsorption of the clove extract constituents at the active sites of steel surface hindering the corrosion reaction. Table 1 shows also that the cathodic Tafel slope vary slightly with the change of the concentration of the extract which indicates that the absorbed constituents of the extract only blocking the surface of the steel without change the mechanism of the corrosion reaction.

The above results indicate that clove extract act as a good green inhibitor for the general corrosion of stainless steel 304.

Table 1. The electrochemical polarization parameters of stainless steel 304 in 0.5 M HCl solution in absence and presence of different concentrations of clove extract.

Extract conc. (ppm)	$-E_{\text{corr}}$ (mV vs. SCE)	$-E_p$ (mV vs. SCE)	R_p (Ohm·cm ²)	i_{pass} (mA·cm ⁻²)	$-\beta_c$ (mV·decade ⁻¹)	i_{corr} (mA·cm ⁻²)	%P
0.0	395	166	172	1460	141	1703.2	–
2.0	387	142	187	897	140	1049.4	38.4
4.0	362	136	201	667	146	579.2	66.0
6.0	346	121	227	418	182	421.4	75.3
8.0	335	116	269	244	144	314.0	81.6
10.0	317	109	284	157	138	225.3	86.8
12.0	311	104	291	72	141	186.8	89.1

Table 1 shows also that the increase of the concentrations of the clove extract in the acid solution leads to: 1) markedly decrease of the passivating current (i_{pass}), 2) markedly increase of the polarization resistance (R_p) and 3) shift of the pitting potential (E_p) to more noble potentials. These results indicate that clove extract act also as a good green inhibitor for pitting corrosion of stainless steel 304 in 0.5 M HCl.

III.2. Electrochemical impedance spectroscopy (EIS) results

Nyquist impedance plots of stainless steel in 0.5 M HCl, without and with different concentrations of clove extract are presented in Figure 3. The plots consist of distorted semicircles followed by diffusion tail indicative that the corrosion process takes place under diffusion control. The increase in the size of the semicircle in presence of the clove extract indicates that a barrier gradually forms on the stainless steel surface. The impedance spectra were analyzed by fitting the experimental data to the equivalent circuit model shown in Figure 4 [20, 21]. The protection efficiency of the extract was calculated from the impedance measurements using the relation:

$$\%P = [(R_{\text{ct}} - R_{\text{ct}_0}) / R_{\text{ct}}] \cdot 100 \quad (1)$$

Where R_{ct} and R_{ct_0} are the charge transfer resistances, in absence and presence of clove extract respectively.

Figure 3. Nyquist plots for stainless steel 304 in 0.5 M HCl solution in absence and presence of different concentrations of clove extract.

Figure 4. Schematic for the equivalent circuit of stainless steel 304.

Figure 5. Computer fitting of equivalent circuit of Nyquist and Bode plots for stainless steel 304 in 0.5 M HCl in presence of 4 ppm clove extract.

Figure 5 shows the computer fitting of the measured data for Nyquist and Bode plots for stainless steel 304 in 0.5 M HCl in presence of 4 ppm clove extract. Fitting the EIS data to the equivalent circuit model revealed a good agreement between fitted and measured spectra. The values of the electrochemical parameters obtained from EIS for stainless steel in 0.5 M HCl solution containing different clove extract concentrations and the inhibition efficiency are given in Table 2. It is clearly seen that the presence of clove extract enhances the values of each of the charge transfer resistance R_{ct} and protection efficiency % P of the extract. However, the values of double layer capacitance Q_{dl} decrease with increasing the concentration of the extract due to its adsorption at stainless steel /solution interface.

Table 2. Values of the electrochemical impedance parameters for stainless steel 304 in 0.5 M HCl solution in absence and presence of different concentrations of clove extract at 30°C.

Conc. (ppm)	R_s	Q_f ($\mu F \cdot cm^{-2}$)	n_1	R_p ($Ohm \cdot cm^2$)	R_{ct} ($Ohm \cdot cm^2$)	Q_{dl} ($\mu F \cdot cm^{-1}$)	n_2	R_2 ($Ohm \cdot cm^2$)	Q_3 ($\mu F \cdot cm^{-2}$)	n_3	% P
0.0	0.2	57	0.9	166	123	1387	0.6	212	4688	0.6	0.0
2.0	0.1	54	0.9	172	201	940	0.6	344	4374	0.6	38.8
4.0	0.2	52	0.9	184	371	827	0.6	468	4289	0.6	66.8
6.0	0.3	48	0.9	195	504	734	0.6	401	4088	0.6	75.6
8.0	0.2	43	0.9	213	687	622	0.6	845	3753	0.6	82.1
10.0	0.2	38	0.9	237	994	537	0.6	1108	3420	0.6	87.6
12.0	0.2	35	0.9	259	1275	457	0.6	1170	3106	0.6	90.4

III.3. Mass loss results

Figure 6 shows the variation of the weight loss during 72 hours at 30°C for stainless steel in 0.5 M HCl in absence and presence of different concentrations of clove extract. It is obvious that weight loss increases linearly with time irrespective of the extract concentrations, the slope of the lines which represents the corrosion rate R decreased with the increase of the extract concentration. The percentage inhibition efficiency (% P) was calculated using the relation:

$$\% P = [(R_0 - R)/R_0] \cdot 100 \quad (2)$$

Where R_0 and R are the corrosion rate in $g \cdot cm^{-2} \cdot hr^{-1}$, in absence and presence of extract.

Figure 6. Time evolution of the weight loss experienced by stainless steel 304 samples in 0.5 M HCl solutions containing different concentrations of clove extract at 30°C.

Table 3. Corrosion parameters obtained from weight loss of stainless steel 304 in 0.5 M HCl solutions containing different concentrations of clove extract at 30°C.

Conc. (ppm)	Rate ($\text{g}\cdot\text{cm}^{-2}\cdot\text{hr}^{-1}$)	%P
0	0.01143	—
1	0.01002	12.3
2	0.00720	37.0
4	0.00400	65.0
6	0.00289	74.7
8	0.00224	80.4
10	0.00165	85.6
12	0.00135	88.2

The results obtained at different extract concentrations are summarized in Table 3. Higher concentrations of the extract led to a decrease in the values of the corrosion rate, while the inhibition efficiencies increased. These results indicate that increasing the concentration of the extract in the HCl solution leads to greater surface coverage by the extract molecules that affords corrosion protection to the stainless steel substrate.

Figure 7 shows the variation of %*P* of the clove extract calculated from the results of polarization, impedance and weight loss with its concentration in 0.5 M HCl solution. It is clear that a good agreement between the data obtained from the different techniques.

Figure 7. Variation of %*P* of clove extract in 0.5 M HCl solution with its concentration using mass loss, potentiodynamic polarization and electrochemical impedance spectroscopy techniques.

III.4. Optical microscopy results

Optical microscopy was employed to examine the surface modification of the stainless steel samples that had been immersed in 0.5 M HCl solution, both in absence and presence of the clove extract, for 72 hours.

Figure 8 shows optical micrographic photo of stainless steel in 0.5 M HCl free from or containing clove extract after 72 hours. As seen from the micrographic photos, the scratch marks of the emery paper still viewed within the experimental exposure time for stainless steel that was immersed in the acid solution containing 10 ppm clove extract whereas in the medium free from the extract, the scratch marks of stainless steel disappeared due to severe corrosion.

Figure 8. Optical micrographic photos (40x) of stainless steel in 0.5 M HCl, in absence and presence of clove extract.

III.5. Application of adsorption isotherms

Langmuir, Flory–Huggins isotherms and kinetic–thermodynamic model [22] were used to fit the adsorption data of the cloves extract.

The Langmuir isotherm is given by

$$[\theta/(1-\theta)] = K[C] \quad (3)$$

Where K is the binding constant representing the interaction of the additives with metal surface and C is the concentration of the additive.

Flory–Huggins isotherm is given by

$$\theta/[x(1-\theta)^x] = K[C] \quad (4)$$

Where x is the size parameter and is a measure of the number of adsorbed water molecules substituted by a given cloves molecule.

And the kinetic–thermodynamic model is given by

$$\log[\theta/(1-\theta)] = \log K' + y \log C \quad (5)$$

Where y is the number of the adsorbed molecules of the ingredient of clove extract occupying one active site. The binding constant K is given by:

$$K = K'^{(1/y)} \quad (6)$$

Figure 9. Linear fitting of the data of adsorption of clove extract to Langmuir isotherm for stainless steel 304 in 0.5 M HCl solution.

Figure 10. Linear fitting of the data of adsorption of clove extract to Flory–Huggins isotherm for stainless steel 304 in 0.5 M HCl solution.

Figure 11. Linear fitting of the data of adsorption of clove extract to kinetic–thermodynamic model for stainless steel 304 in 0.5 M HCl solution.

Figures (9–11) show the application of the above-mentioned models to the data of clove extract obtained from impedance measurements. The parameters obtained from the figures are depicted in Table 3.

Table 3. Linear fitting parameters of the data of cloves extract to the used models.

Clove extract	Langmuir	Flory–Huggins		Kinetic–thermodynamic	
	–	x	K	$1/y$	K
From impedance	–	0.54	0.50	0.68	0.37

It is clear that Langmuir isotherm is unsuitable to fit the data indicating that the adsorption process of clove extract on stainless steel surface is non ideal [23–25]. On the other hand, Flory–Huggins isotherm and kinetic–thermodynamic model are found to be applicable. The value of Flory–Huggins size parameter $x \approx 0.5$ indicates that two clove molecules displace one adsorbed water molecule from the stainless steel surface. According of the kinetic–thermodynamic model the term $1/y$ represents the number of active sites occupied by one inhibitor molecule. The table gives $1/y \approx 0.5$ indicating that $y = 2$ and two extract molecules occupy one active site of the stainless steel surface, *i.e.*, these is a multilayer adsorption. The table shows also that is a considerable agreement between the values of the binding constant K calculated from each of the Flory–Huggins isotherm and the kinetic–thermodynamic model.

Conclusion

The results of this study show that:

1. Weight loss, potentiodynamic polarization, electrochemical impedance spectroscopy and optical microscopic results indicated that clove extract act as highly efficient inhibitor for the pitting corrosion of stainless steel in 0.5 M HCl with percent inhibition about 90% at 12 ppm extract.
2. Potentiodynamic polarization results indicated the clove act as anodic inhibitor.
3. The percentage inhibition of the clove extract measured from the three techniques showed a good agreement.
4. Applications of the adsorption isotherms to fit the experimental results indicated that Langmuir isotherm is not applicable and adsorption process is non ideal, Flory–Huggins isotherm and the kinetic–thermodynamic model are applicable and the results indicated that the adsorption process is multilayer.

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