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Evaluation of Aloe vera Gel extract as eco-friendly corrosion inhibitor for carbon steel in 1.0 M HCl

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Abstract

The corrosion inhibition of carbon steel in 1.0 M HCl using the Aloes leaves extract has been studied. Three electrochemical different techniques have been utilized for studying the corrosion inhibition efficiency behavior of Aloe vera Gel as green corrosion inhibitor in addition to Scanning electron microscopy- Energy dispersive X-ray analysis (SEM-EDX) technique. The four techniques which are potentiodynamic polarization, Electrochemical impedance spectroscopy (EIS), Electrochemical frequency modulation (EFM) and SEM-EDX were used for evaluation at room temperature 25 °C. The results show a good efficiency with low concentration of inhibitor, where the electrochemical methods show that 52 % efficiency with low concentration 100 ppm, in addition to surface morphology of carbon steel show a protected surface compared with noninhibited specimen due to formation of inhibitor layers on surface. EDX show that more concentration of chemical elements of gel extract chemical components adsorbed on inhibited specimen compared with non-inhibited specimen. The inhibition efficiency increases with the increasing of the aloe vera gel concentration.

Key Words: Corrosion, Carbon steel, green corrosion inhibitor, Aloe vera, gel.

1. INTRODUCTION

Carbon steel is used in many industries around the world due to: low cost, properties, ease of fabrication, availability, weldability. Carbon steel materials are used in Petroleum applications such as pressure vessels, heat exchangers, boilers, pipelines, casing and other facilities because of their good strength and ductility [1-3]. Cost of corrosion obliges countries and companies to invest in corrosion behavior and how to reduce this impact. A lot of researches and papers are published by corrosion scientists, many of corrosion inhibitors are toxic and harms the environment, so the modern papers interests in green corrosion inhibitors and study of their effect on corrosion [4-6].

In industry, we use corrosive media such as hydrochloric acid in pickling to remove rust from steel and scales from boilers, in oil and gas production we use HCl to acidify an oil production well to dissolve carbonate reservoirs or combined with other chemical compounds to dissolve, clay sand and quartz from rocks. Corrosion inhibitors can be introduced to the well to prevent the acid from breaking down steel casing [7-8].

A lot of organic compounds tend to adsorb on metal surfaces, and play a role of the corrosion inhibition effect, related to: the metal electrostatic attraction on the charged inhibitor molecules; the interaction of lone pair electrons of N, S, P, O or/and double bonds with the vacant d-orbital of the metal; or a combination of some other mechanisms [9]. Plant extracts are mixed organic inhibitors may be used rather than toxic inhibitors and give excellent effect, these extracts are effective, abundant, cheaper and eco-friendly corrosion inhibitors [10-12]. There are more than two hundred different types of chemical compounds in aloe vera. The aloe vera leaf gel contains about 98 to 99% water. Total solid content of the extracted gel is about 0.66% and soluble solids are 0.56% [13]. On dry matter basis

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extracted gel consists of polysaccharides 55 %, sugars 17 %, minerals 16 %, proteins 7 %, lipids 4 % and phenols 1 %. The major polysaccharide is acemannan, the A.Vera gel contains several important vitamins such as vitamins E, C and A. Vitamin B1 (thiamine), niacin, Vitamin B2 (riboflavin), choline and folic acid are also presented in **figure 1**, Some authors also suggested the presence of vitamins B12 (cyanocobalamin) in trace amounts [14-17]. There are several researches in aloe vera as medical extract as shown in previous papers, other papers are published in corrosion science such as The Study of Aloe-Vera gel Extract Inhibitor for Mild Steel in Acetic acid [18]. The effect of Aloe vera extract on corrosion of zinc in HCl solution [19].

The work aimed to extract aloe vera gel as ecofriendly inhibitor to act as an effective corrosion inhibitor protecting the carbon steel from corrosion in the acidic environments. The inhibiting effect of the extract has been assessed using four methods which are potentiodynamic polarization, Electrochemical impedance spectroscopy (EIS), Electrochemical frequency modulation (EFM) and SEM-EDX.



Fig. 1 major components of Aloe vera gel

2. MATERIALS AND METHODS

2.1. Materials

Tests were performed on API 5L grade B carbon-steel. The specifications of API 5L C-Steel have generally been used as the main construction material for flow lines, and transmission pipelines in the petroleum industry [20,21], The chemical

composition of carbon steel (weight %) was Carbon (0.28 %), Manganese (0.81 %), Chromium (0.36 %), Molybdenum (0.10 %), Phosphorus (0.030 %), Sulfur (0.030 %), Nickel (0.33%) and the balance is Fe.

2.2. Solutions

The aggressive solution 1.0 M HCl was prepared by appropriate dilution of analytical grade 37.0 % HCl manufactured by Fisher Chemical with checked distilled water.

2.3. Preparation of plant extract

Stock solution of Aloe vera gel was squeezed out of 500-gram fresh leaves and sieved to obtain 100 ml a clear liquid. To determine the solid content of aloe vera gel, 10.00 gram of gel was dried in oven at temperature 40 °C till mass constant 0.2 gram. The gel was diluted with appropriate quantity of 1.0 M HCl solution to obtain inhibitor test solutions of 20, 40, 60, 80 and 100 ppm concentrations [19].

2.4. Electrochemical methods

The evaluation of the A.Vera Gel corrosion inhibitor has been performed utilizing three different (i) Potentiodynamic techniques which are: Polarization method, (ii) Electrochemical impedance (EIS) and (iii) Electrochemical frequency modulation (EFM). The Apparatus Gamry Potentiostat/galvanostat/ZRA (model Reference 3000) was used for evaluating the A.Vera Gel extract corrosion inhibitor electrically. The apparatus comprises a Gamry framework system version 7.8.2. All measurements were carried out by Ag/AgCl Reference electrode, Graphite counter electrode and C-steel as working electrode having surface area 6.28 cm2 in 1.0 M HCl in the absence and presence of different concentrations (20, 40, 60, 80 and 100 ppm) of the Aloe vera gel inhibitor.

2.5. Surface morphology

Scanning electron microscope (SEM) and Energy dispersive X-ray (EDX) methods are utilized to confirm the results obtained from electrochemical methods.

3. RESULTS AND DISCUSSION

3.1. Structure confirmation using FTIR-Spectroscopy

The chemical structure of the mixed compounds of A.Vera Gel was mentioned in previous introduction confirmed using Fourier Transmission Infra Red (FTIR) spectroscopy as shown in figure 2. Absorption band 3418 cm⁻¹ refer to -OH alcohol and intermolecular bonds, while 2926 cm⁻¹ is CH alkane

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and OH intramolecular bonds, 1729 cm⁻¹ C=O refer to aldehyde and esters, 1622 cm⁻¹ indicate to NH amines and C=C as conjugated alkene, aromatic compounds and unsaturated ketone, 1431 cm⁻¹ COOH, 1251 cm⁻¹ refer to aromatic ester and amines, 1078 cm⁻¹ amines, primary alcohol and alkoxy compounds, 1035 cm⁻¹ refer to sulfoxides or amine, while 787 cm⁻¹ indicate to alkenes [22-24].



Fig. 2 FTIR spectra of Aloe Vera Gel

3.2. Potentiodynamic polarization technique

The polarization behavior of carbon steel immersed in 1.0 M HCl in the absence and presence of varying concentrations of A.Vera gel inhibitor at 25 °C was shown in Fig.3 Electrochemical parameters such as corrosion potential (E_{corr}), cathodic and anodic Tafel slopes (β_a , β_c) were calculated, also the corrosion current density (I_{corr}) and the percentage inhibition efficiency (IE%) were calculated and listed in Table 1. The values of surface coverage degree (θ) and the percentage inhibition efficiency (IE%) are calculated using this Equation [25,26]:

$$IE\% = \theta x \ 100 = \left(\frac{I_{corr}^{\circ} - I_{corr}}{I_{corr}^{\circ}}\right) x \ 100$$

where $I_{o_{corr}}$ and I_{corr} is the values of corrosion current density in the absence and presence of inhibitors. The experimental data refer to decreasing corrosion current densities (I_{corr}) values with increasing concentration of Aloe vera gel inhibitor with respect to the blank curve. Also, it is clear that the values of corrosion potential (E_{corr}) were almost constant. This indicated that the Aloe vera gel inhibitors act as mixed type inhibitors. Furthermore, the slopes of the cathodic and anodic Tafel lines are approximately constant and independent on the inhibitor concentration. This means that the A.vera gel inhibitors have an effect on the corrosion rate while they do not effect on the metal dissolution mechanism [27,28].

Table 1 Electrochemical parameters obtained by Potentiodynamic polarization technique for carbon steel in absence and presence of various concentrations of aloe vera Gel plant extract in 1.0 M HCl at 25 °C

| | Conc. | - E _{corr} | I _{corr} | R _p | β_a | β_{c} | CR | IE | θ |
|-------|-------|---------------------|---------------------|-------------------------|------------|-------------|-------|------|-------|
| | (ppm) | (mV) | mA.cm ⁻² | $(\Omega \text{ cm}^2)$ | (V/decade) | (V/decade) | (mpy) | % | |
| Blank | | 383 | 3.440 | 10.81 | 0.124 | 0.277 | 250.6 | | |
| | 20 | 381 | 3.140 | 12.09 | 0.125 | 0.291 | 228.2 | 8.9 | 0.087 |
| | 40 | 379 | 3.000 | 15.21 | 0.155 | 0.326 | 218.3 | 12.9 | 0.128 |
| Gel | 60 | 378 | 2.770 | 16.39 | 0.153 | 0.330 | 201.4 | 19.6 | 0.195 |
| | 80 | 379 | 2.620 | 15.57 | 0.136 | 0.304 | 190.7 | 23.9 | 0.238 |
| | 100 | 391 | 1.990 | 17.27 | 0.112 | 0.270 | 144.6 | 42.3 | 0.422 |



Fig.3 Potentiodynamic polarization curves for corrosion of C-Steel in 1.0 M HCl in the absence and presence of different concentrations of the aloe vera gel at 25 °C

3.3. Electrochemical impedance spectroscopy (EIS)

Figure 4 show the obtained Nyquist graph for carbon steel in 1.0 M HCl solutions without and with various concentrations of the Aloe vera gel inhibitor at 25 °C. Nyquist plot revealed that the impedance response of carbon steel shows a significant change after the addition of the inhibitor in 1.0 M HCl solution.



Fig. 4 The Nyquist plot for corrosion of C-Steel in 1.0 M HCl in the absence and presence of different concentrations of Aloe vera gel inhibitor at 25 °C.

By Comparing between the semicircle radii of the carbon steel in uninhibited solution and these in the presence of different concentrations of Aloe vera gel inhibitor showed that the real axis intercept at low frequencies in the presence of extracted A.Vera gel inhibitor is larger than that in the absence of the Aloe vera gel (blank) and increases with increasing gel inhibitor concentration. This confirms that the impedance of carbon steel corrosion, increased with the increase in the inhibitor concentrations in 1.0 M HCl solution. The impedance of C-Steel corrosion increases result to the increasing of surface coverage (θ) of inhibitor molecules on the surface of working C-Steel electrode, which results in an increase in the inhibition efficiency. Adsorbed inhibitor molecules form a protective film, which isolate the carbon steel surfaces and inhibits both cathodic and anodic reactions at the steel surface [29,30]. EIS graph of the aloe vera gel inhibitor were analyzed using a suitable equivalent circuit (EC) as shown in Fig. 5 where, Rs is the solution resistance, R_{ct} is the charge transfer resistance, C_{dl} is the electrochemical double layer capacitance. The electrochemical impedance parameters such as charge transfer resistance (R_{ct}), double layer capacitance (C_{dl}) , and inhibition efficiency (IE %), were calculated and listed in Table 2.

The inhibition efficiencies and the surface coverage (θ) are obtained from the impedance measurements were adopted from the literature [12,31] defined by the following relation:

$$IE\% = \theta x \ 100 = \left[1 - \left(\frac{R_{ct}}{R_{ct}}\right)\right] x \ 100$$

where R^{o}_{ct} and R_{ct} are the charge transfer resistance in the absence and presence of inhibitor, respectively.

Table 2 indicate that by increasing the inhibitor concentrations, R_{ct} values increased significantly. The inhibition efficiencies, calculated from EIS show the same trend as those obtained from polarization, the difference of inhibition efficiency from the two

methods may be attributed to the difference surface status of the working electrode in the three measurements.

Table 2 EIS parameters of C-Steel in 1.0 M HCl in the absence and presence of different concentrations of Aloe vera gel at 25°C.

| Inh. | Conc. ppm | R_s Ωcm^2 | $\frac{R_{p}(R_{ct})}{\Omega cm^{2}}$ | n | Yo x 10^{3} Ω^{-1} cm ⁻² s ⁿ | C _{dl} F cm- ² | IE % | θ |
|-------|--------------|------------------------|---------------------------------------|-------|----------------------------------------------------------------|---------------------------------------|---------|--------|
| Blank | | 0.488 | 10 | 0.971 | 1.083 | 1429 | | 0 |
| | 20 | 0.495 | 11.7 | 0.859 | 0.982 | 4556 | 14.53 | 0.1453 |
| | 40 | 0.511 | 14.1 | 0.869 | 0.902 | 3749 | 29.08 | 0.2908 |
| Gel | 60 | 0.653 | 16.8 | 0.863 | 0.88 | 4040 | 40.48 | 0.4048 |
| U | 80 | 0.582 | 19.1 | 0.883 | 0.893 | 3248 | 47.64 | 0.4764 |
| | 100 | 0.788 | 20.9 | 0.85 | 1.027 | 5970 | 52.15 | 0.5215 |



Fig.5 Electrical equivalent circuit model used to fit the impedance spectra data.

3.4. Electrochemical frequency modulation (EFM)

In the EFM method, a potential disturbance by two sine waves of distinct frequencies is utilized to the system. The alternating current response which is coming from this disturbance consists of current peaks of different frequencies as shown in figure 6, these peaks at the intermodulation frequencies can reveal the corrosion rate and Tafel parameter, also were calculated and listed in Table 3 [32,33].

The EFM technique is a powerful method due to the causality factors which act as an internal check on the quality of the EFM measurement, the causality factor is calculated of the current react and listed in Table 3. The theoretical values of causality factors are 2 and 3, so if the causality factors differ from these values, it may be said that the measurements are under the effect of the noise. When the causality factors are approximately 2 and 3, there is a causal relationship between the disturbance signal and the response signal. Then the data are assumed to be reliable [32,34]. The spectrum contains current responses assigned for harmonical and intermodulation current peaks. The larger peaks were utilized for calculation of the corrosion current. IE % EFM was calculated using previous equation [35-37].

It is clear that the corrosion current density values decrease by increasing the concentration and the inhibition efficiency increase by increasing inhibitor concentration as demonstrated in Table 3. The data obtained from EFM are in good agreement with the results those obtained from polarization and EIS.

| | Conc. | Icorr | βa | βc | CR | IE | CE-2 | CE-3 |
|-----|-------|---------------------|-------------|-------------|-------|-------|-------|-------|
| | (ppm) | mA.cm ⁻² | (mV/decade) | (mV/decade) | (mpy) | % | CI 2 | CI J |
| | Blank | 2.116 | 89.17 | 131.6 | 154.0 | ••••• | 1.988 | 3.382 |
| | 20 | 1.612 | 80.61 | 126.8 | 117.3 | 23.8 | 1.980 | 3.203 |
| | 40 | 1.524 | 89.63 | 138.7 | 110.9 | 27.9 | 2.022 | 3.349 |
| Gel | 60 | 1.339 | 96.38 | 114.9 | 97.41 | 36.7 | 1.919 | 3.299 |
| | 80 | 1.191 | 91.53 | 112.7 | 86.63 | 43.7 | 1.945 | 3.287 |
| | 100 | 1.048 | 26.81 | 33.27 | 76.27 | 50.4 | 2.414 | 3.300 |

Table 3 Electrochemical parameters obtained by EFM technique for carbon steel in absence and presence of various concentrations of Aloe vera Gel inhibitor in 1.0 M HCl at 25°C

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| Medium | Composition (wt%) | | | | | | | | |
|-----------------------------------------|-------------------|-------|------|------|------|------|------|--|--|
| | Fe | 0 | С | Ν | S | Р | Mn | | |
| (a) Carbon steel | 97.01 | 1.84 | 0.28 | 0.00 | 0.03 | 0.03 | 0.81 | | |
| (b) C-Steel in 1.0 M HCl | 81.27 | 11.96 | 5.31 | 0.21 | 0.27 | 0.18 | 0.80 | | |
| (c) C-Steel in 1.0 M HCl with inhibitor | 85.03 | 4.05 | 8.13 | 1.34 | 0.35 | 0.23 | 0.87 | | |

Table 4 EDX analysis results of C-Steel specimens alone and in 1.0 M HCl in the absence and presence of inhibitor



Fig.6 EFM spectra for corrosion of C-Steel in 1.0 M HCl in the absence and presence of different concentrations of the Aloe vera gel at 25 °C

3.5. Characterization of Surface Morphology

3.5.1. Scanning electron microscopy (SEM)

The SEM micrographs of carbon steel surface before and after exposing to corrosive acidic solution in the absence and presence of inhibitor are shown in Fig. 7. Figure 7-a shows the abraded c-steel surface before immersion in corrosive solution and Fig. 7-b, c reveals the SEM images of steel surface immersed in 1.0 M HCl solution for 24-hr. in the absence and presence of gel inhibitor. From Fig. 7-b, it is obviously seen that the metal surface was severely corroded due to presence of corrosive media. However, the appearance of c-steel surface is different after the addition of the inhibitor solution, as shown in Fig. 7-c. The protective layer of inhibitor is adsorbed on the c-steel surface and prevents further corrosion [38].

3.5.2. Energy dispersive x-ray analysis (EDX)

EDX spectra Fig. 8 were recorded to determine the percentage of oxygen, carbon and nitrogen in c-steel in the absence and presence of inhibitor. The EDX analysis of c-steel in 1.0 M HCl in the absence and presence of inhibitor refer to the c-steel in the aggressive solution with inhibitor (Fig. 8-c) had high carbon and nitrogen concentration (Table 4), while in Fig. 8-b are lower, due to adsorption of hetero-cyclic organic compounds with nitrogen elements. In addition, a high oxygen concentration of fig. 8-b was observed in the absence of inhibitor due to forming of iron oxides, while in (c) is lower. These values confirm the formation of a protective layer by inhibitor molecules on the carbon steel surface [39,40].

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Fig.7 SEM images of (**a**) carbon steel, (**b**) carbon steel in 1.0 M HCl without addition of inhibitor and (**c**) carbon steel in 1.0 M HCl with in the presence of 100 ppm Gel inhibitor.



Fig.8 EDX spectrum of (**a**) carbon steel, (**b**) carbon steel in 1.0 M HCl without addition of inhibitor and (**c**) carbon steel in 1.0 M HCl with in the presence of 100 ppm Gel inhibitor.

4. CONCLUSIONS

An eco-friendly corrosion inhibitor was extracted and investigated to inhibit the corrosion of C-steel in 1.0 M HCl. The Aloe vera gel exhibit good inhibition with low concentration. The inhibition efficiency of aloe vera gel extract increases with

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increasing the concentration at 25 °C. Polarization data show that the investigated extracts act as a mixed-type inhibitor in 1.0 M HCl. EIS spectra exhibit individual capacitive loop. The presence of inhibitor in 1.0 M HCl solutions increases R_{ct} . The three electrochemical techniques give the same results with few neglected deviations. The SEM-EDX refer to good inhibition of aloe vera gel as corrosion inhibitor.

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