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INHIBITION OF CORROSION OF CARBON STEEL IN HYDROCHLORIC ACID BY AQUEOUS LEAVES EXTRACT OF *OXALIS CORNICULATA LINN* (OCLP)

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ABSTRACT

The corrosion inhibition of carbon steel is sunken in 0.5N hydrochloric acid has been evaluated at room temperature by using the mass loss method. The corrosion rate and inhibition efficiency have been attained from the mass loss method. The efficiency of corrosion inhibition improves when the content of an inhibitor, specifically aqueous leaves extract of the *oxalis corniculata linn* plant, is increased. When the concentration of inhibitor is increased, the corrosion rate reduces. This is because a protective coating forms over the carbon steel surface at greater concentrations of inhibitor solution, which prevents the active site of carbon steel from becoming active. The creation of a protective coating over the carbon steel surface was investigated using electrochemical tests. Surface analysis techniques such as scanning electron microscopy can also detect it. Smoothness and roughness of carbon steel surface like polished, corroded and inhibitor system has been appraised by SEM.

Keywords: Acidic solutions, Corrosion, Carbon steel, Mass loss method, *oxalis corniculata linn* plant and Scanning Electron Microscopy

INTRODUCTION

Since the industrial insurrection for industrial and structural applications, carbon steel is widely used in machinery and automobile manufacturing. Carbon steel beams, like any structural steel beam, offer a tremendous amount of strength and can be utilized as a construction frame material. It is inexpensive, versatile in terms of cutting and coating processes, and has good weldability while maintaining adequate physical qualities. Crude oil refining took place in a variety of caustic circumstances. Because of these characteristics, it can be used to build frames, panels, and other similar items. Carbon steel is also resistant to earthquakes and wind. Acidic media are essential in the investigation of carbon steel corrosion because of their industrial applications, which include industrial cleaning, acid pickling, acid descaling, petrochemical processes, and oil-well acid in oil recovery [1-3]. The major problems in the industrial use of acids are corrosion of the metal equipment, contamination of the circulating acid leads to the damage of types of equipment. Generally, acid-using materials undergo corrosion and it is an unavoidable one. The corroded materials besides loss in weight and cross-section can lead to hostile effects on the material properties. Therefore,

it is required to prevent or reduce it by using inhibitors or additives. Corrosion inhibitors are used in the industrial process to control metal dissolution, especially in acid, neutral and base environments. Some organic compounds containing electron-donating groups or polar functional groups, heteroatoms, aromatic rings with π- electrons are extensively used as effective corrosion inhibitors in controlling the corrosion. These inhibitors are chemically or physically deposited on the metal surface, providing a blanket that isolates the metal from the corrosive ions present in the medium. Organic compounds with hetero atoms, such as O, N, S, and numerous bonds, make up the majority of well-known inhibitors. Corrosion has a wide range of implications, and the effects on the safe, dependable, and efficient functioning of equipment or structures are frequently more catastrophic than mere material loss [4-6]. Several researchers investigated the effects of organic compounds on the corrosion of copper, aluminum alloys, mild steel, carbon steel, and composites in acidic, alkaline, and neutral conditions [7-8]. Because the majority of these organic chemicals are not only expensive but also harmful to humans and the environment, their use as corrosion

inhibitors is restricted. As a result, attempts have been made to produce corrosion inhibitors that are both cost-effective and non-toxic. Plant extracts are thought to be an extraordinarily rich source of corrosion inhibitors that are safe for the environment **[9-10]**. Traditional medicine has employed *oxalis corniculata linn*, a member of the Leguminosae family, for therapeutic purposes. The plant is commonly used for human consumption as fodder and green vegetables. However, a comprehensive study of its phytochemical profile and pharmacological characteristics is lacking. Antioxidants and cholinesterase inhibitors are found in the plant, which is also utilized for anti-inflammatory, antiseptic, analgesic, antirheumatic pain, and antibacterial reasons **[11]**.

The primary goal of this study is to determine the efficacy of aqueous leaves extracts of the *oxalis corniculata linn* plant as a corrosion inhibitor for carbon steel immersed in 0.5N HCl. The mass loss method was used to analyze the impact of the inhibitor on corrosion rate and inhibition efficiency. Electrochemical research such as alternating current impedance spectra and polarisation studies are used to determine the mechanistic features of corrosion inhibition. The protective film was formed over the surface of carbon steel has been analyzed by Fourier Transform Infra-red (FTIR) spectroscopy technique. The smoothness of carbon steel when compared to polished carbon steel, corroded carbon steel (blank) and carbon steel in an inhibitor system has been characterized by Scanning Electron Microscopy (SEM).

MATERIALS AND METHODS

Corrosion inhibition of carbon steel in the hydrochloric acid medium by aqueous leaves extract of *oxalis corniculata linn* plant has been investigated.

Carbon steel specimens Preparation

Carbon - 2.0 %, Sulphur – 0.026 %, Phosphorus - 0.06 %, Manganese - 0.4 % and the balance iron were polished to mirrors finish and degreased with acetone and used for mass loss method. The solution (0.5N HCl) was prepared by dilution of an analytical grade hydrochloric acid with double distilled water.

Preparation of stock solutions

Double distilled water was used wherever necessary in the preparation of solutions. Analytical grade HCl is taken as such and they were diluted to the required concentration. The required concentration of the aqueous leaves extract of *oxalis corniculata linn* plant stock solution was prepared by dissolving the leaves of *oxalis corniculata linn*

plant in minimum amount of water and making up to the desired volume with double distilled water. Then the required volume from the inhibitor stock solution was added to the hydrochloric acid solution to obtain the desired concentration.

Mass loss method

Mass loss measurements were performed for 3 hours by immersing the carbon steel specimens in 0.5M hydrochloric acid without and with different concentrations (2, 4, 6, 8, 10 ml) of aqueous leaves extract of *oxalis corniculata linn* plant inhibitor.

After the elapsed time, the specimen was taken out, washed, dried and weighed accurately.

The inhibition efficiency (IE %) was determined by the following equation [12]

$$IE\ (\%) = \frac{W_0 - W_i}{W_0} \times 100$$

Where W_i and W_0 are the weight loss values in g in presence and absence of an aqueous leaves extract of *oxalis corniculata linn* plant inhibitor.

Electrochemical Techniques

A PC-controlled electrochemical impedance analyzer model CHI6608 Microcell kit Princeton electrochemical Analyzer system with Electrochemistry software was used to assess electrochemical impedance and potentiodynamic polarisation. These tests were carried out in a traditional three-cell electrode cell with a total volume of 100 ml. A saturated calomel electrode and a cylindrical-shaped graphite electrode were employed as the reference and counter electrodes, respectively. A PC-controlled electrochemical impedance analyzer model CHI6608 Microcell kit Princeton electrochemical Analyzer system with Electrochemistry software was used to assess electrochemical impedance and potentiodynamic polarisation. These tests were carried out in a traditional three-cell electrode cell with a total volume of 100 ml. A saturated calomel electrode and a cylindrical-shaped graphite electrode were employed as the reference and counter electrodes, respectively. Before each run, the carbon steel working electrode was abraded and washed with several grades of emery sheets. This working electrode had a surface area of 3.14 cm2. To perform the OCP test, the working electrode was immersed in both uncontrolled and inhibited acidic solutions. During polarisation measurements in both the anodic and cathodic orientations, the OCP (Eocp) caused a shift in potential. The potential scan rate was 0.17 mV s–1. To give Eocp adequate time to stabilize, the electrode

was submerged in the tested solution for 30 minutes **[13]**.

Surface Examination Techniques

For three hours, the carbon steel specimens were immersed in blank and aqueous leaves extract of *oxalis corniculata linn* plant inhibitor solutions. The specimens were taken out and dried after 3 hours. Various analysis techniques were used to investigate the nature of the film that formed on the surface of the carbon steel specimens.

Surface analysis by FTIR spectra

A Perkin–Elmer 1600 spectrophotometer was used to record FTIR spectra. The film was carefully removed, and the FTIR spectra were taken after it was thoroughly combined with KBr and formed into pellets. The specimens were taken out of the test fluids and dried after a 3-hour immersion period in varied settings. The coating that had formed on the surface was carefully scratched and properly mixed to ensure that it was consistent throughout **[14]**. A Perkin–Elmer1600 FTIR spectrophotometer with a resolving power of 4 cm^{-1} was used to record the FTIR spectra of the powder (KBr pellet).

Scanning Electron Microscopic studies (SEM)

SEM was utilized to look at the difference like the metal surface before and after it came into direct contact with the corrodent solution, as well as the influence of the inhibitor **[15]**. As a result, SEM was utilized to examine the topography of the mild steel surface after it had corroded in both the presence and absence of the inhibitor. The SEM image was captured using a JEOL MODEL JSM 6390 SEM equipment.

RESULTS

Results of mass loss method

In the presence and absence of different doses of an aqueous leaves extract of *oxalis corniculata linn* plant inhibitor, mass loss assays were carried out in 0.5N of HCl. The corrosion rates (CR) of carbon steel immersed in 0.5N hydrochloric acid, as well as inhibition efficiencies (IE), were measured using the mass loss method in the absence and presence of aqueous leaves extract of *oxalis corniculata linn* plant inhibitor. **Table 1** shows the inhibitory efficiency and corrosion rate data.

Electrochemical methods

The electrochemical measurements encompass a way of calculating the rate of corrosion of carbon steel. It permits rapid evaluation of the performance of inhibitors,

the durability of surface film and also the inhibition efficiency of corrosion inhibitors.

The following techniques were used for carbon steel corrosion in 0.5M HCl in the absence and in the presence of the OCLPLE inhibitor system to know whether they act as cathodic or anodic or mixed type inhibitors and also to formulate an appropriate mechanism for their inhibition action on the carbon steel corrosion.

Results of potentiodynamic polarization study

The creation of a protective coating on the carbon steel surface was investigated using polarization analysis. The linear polarization resistance values (LPR) increase as a protective coating is developed on the carbon steel surface, while the corrosion current value (I_{corr}) drops. **Figure 1** depicts the potentiodynamic polarization curves of carbon steel immersed in 0.5M HCl in the presence and absence of OCLPLE inhibitor. **Table 2** lists the corrosion characteristics, including corrosion potential (E_{corr}), Tafel slopes, bc and ba, linear polarization resistance (LPR), and corrosion current (I_{corr}).

Results of alternating current impedance spectra

The establishment of a protective layer over the carbon steel surface has been supported using alternating current (AC) impedance spectra (electrochemical impedance spectra). Charge transfer resistance (R_t) increases, double layer capacitance value (C_{dl}) lowers, and impedance log (z/ohm) value increases when a protective layer is developed on the carbon steel surface. **Figure 2** (Nyquist plots), **Figure 3** (Bode plots), and **Figure 4** (AC impedance spectra) of carbon steel immersed in 0.5M HCl in the absence and presence of an OCLPL inhibitor system (Phase angles). Carbon steel was immersed in various solutions and the AC impedance spectra were obtained. **Table 3** lists the AC impedance parameters, including charge transfer resistance (R_t) and double-layer capacitance (C_{dl}).

Surface characterization of carbon steel by SEM

SEM analysis reveals the type of the surface layer in the presence and absence of corrosion inhibitors, as well as the extent of carbon steel corrosion. The carbon steel surface is examined using SEM images. The carbon steel surface is observed using a scanning electron microscope (SEM). The type of the layer generated on the carbon steel surface in the absence and presence of

aqueous leaves extract of OCL plant leaves inhibitor, as well as the level of carbon steel corrosion, are investigated using SEM micrographs. **Figure 5 (a, b, and c)** show SEM images of carbon steel specimens immersed in 0.5M HCl for three hours in the absence and presence of the OCLPLE inhibitor system, respectively.

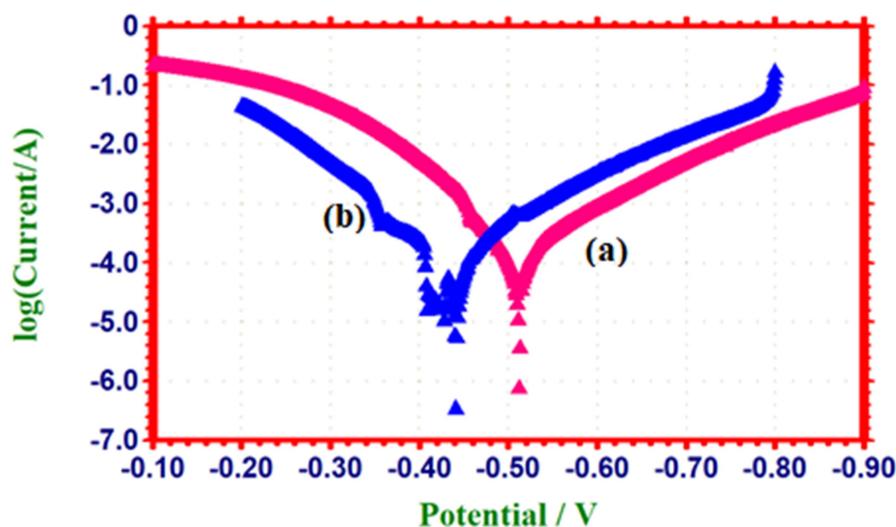For three hours, the carbon steel specimen is immersed in a 0.5M HCl solution containing an inhibitor. The specimen is removed, dried, and examined using a scanning electron microscope (SEM). **Figure 5a** shows photographs of fresh carbon steel (control). **Figure 5b** shows a carbon steel surface that has been degraded by metal dissolving in 0.5M HCl (blank). **Figure 5c** shows a carbon steel surface that has been dipped in an acid-containing inhibitor.

Table 1: Corrosion rates (CR) and inhibition efficiency (IE %) data obtained from mass loss measurements for carbon steel is immersed in 0.5N hydrochloric acid without and with various concentrations of aqueous leaves extract of *oxalis corniculata linn* plant.

➤ **Inhibitor System: Aqueous leaves extract of an *oxalis corniculata linn* plant**
➤ **Immersion period: 3 hours**

Concentration of aqueous leaves extract of OCLP inhibitor (%)	Corrosion rate (mdd)	Inhibition Efficiency (%)
blank	225.10	-
2	78.50	55.62
4	60.63	69.89
6	39.23	85.74
8	28.53	88.91
10	10.70	93.50

Figure 1: Potentiodynamic polarization curves for corrosion of mild steel in 0.5M HCl in absence and presence of TRPLE inhibitor
(a) Mild steel in 0.5M HCl (blank)
(b) Mild steel in 0.5M HCl with 10% aqueous leaves extract of TRP

Table 2: Potentiodynamic Polarization parameters for the corrosion of carbon steel in 0.5M HCl for the aqueous extract of OCLPL system

Concentration of the aqueous leaves extract of OCLP (%V/V)	E_{corr} mV/SCE	Tafel slope		I_{corr} A / cm^2	LPR Ω/cm^2
		ba, mV/dec	bc, mV/dec		
blank	- 512	078	124	1.771×10^{-6}	118.4
10	-441	052	134	9.864×10^{-5}	167.3
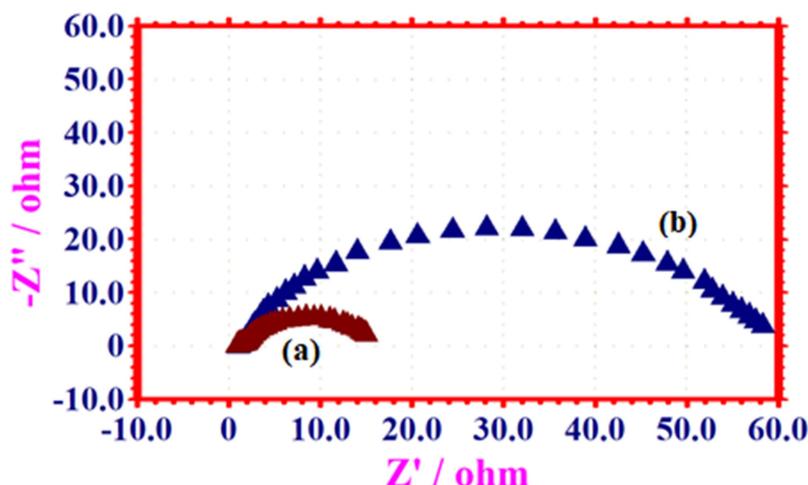
Figure 2: AC impedance spectra of carbon steel immersed in to 0.5M HCl in the absence and presence of OCLPLE inhibitor (Nyquist plots)
(a) Carbon steel in 0.5M HCl without inhibitor
(b) Carbon steel in 0.5M HCl with 10% aqueous leaves extract of OCLP.

Table 3: Electrochemical impedance parameters from Nyquist plots for the corrosion of carbon steel for aqueous leaves extract of TRP leaves in 0.5 M HCl

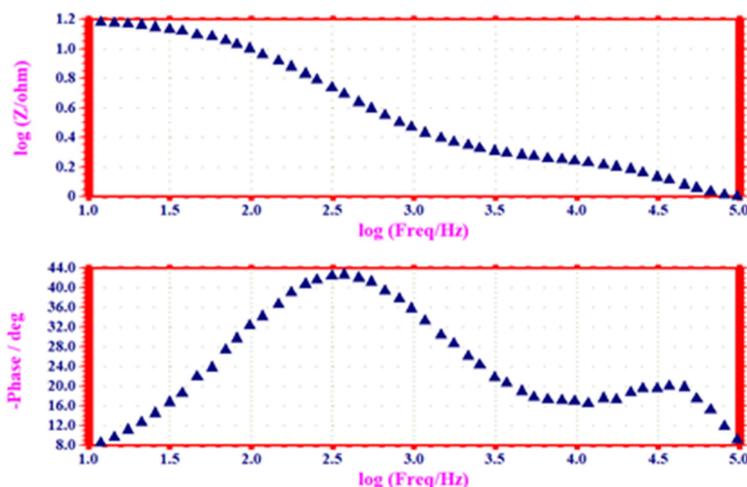| Concentration of the aqueous leaves extract of OCLP (%v/v)	Nyquist plot		Impedance Log (z/ohm)	Phase angle (degree)
	R_t, Ω/cm^2	C_{dl} F/cm^2		
Blank	12.94	1.2727×10^{-7}	1.161	43.5
10	33.058	4.9833×10^{-8}	1.3006	44.7

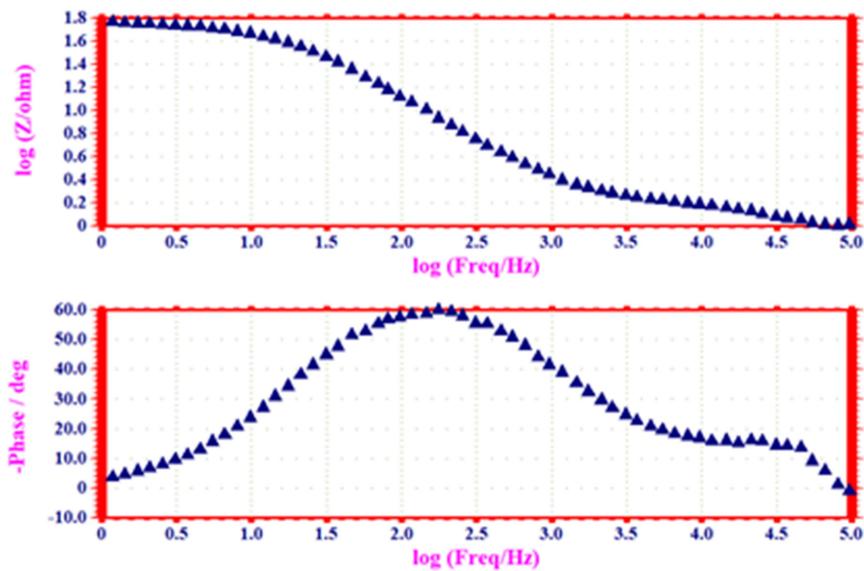**Figure 3a: AC impedance spectra of carbon steel immersed in 0.5M HCl (Bode Plot)**

Figure 3b: AC impedance spectra of carbon steel immersed in 0.5M HCl with 10% aqueous leaves extract of OCLP (Bode Plot)
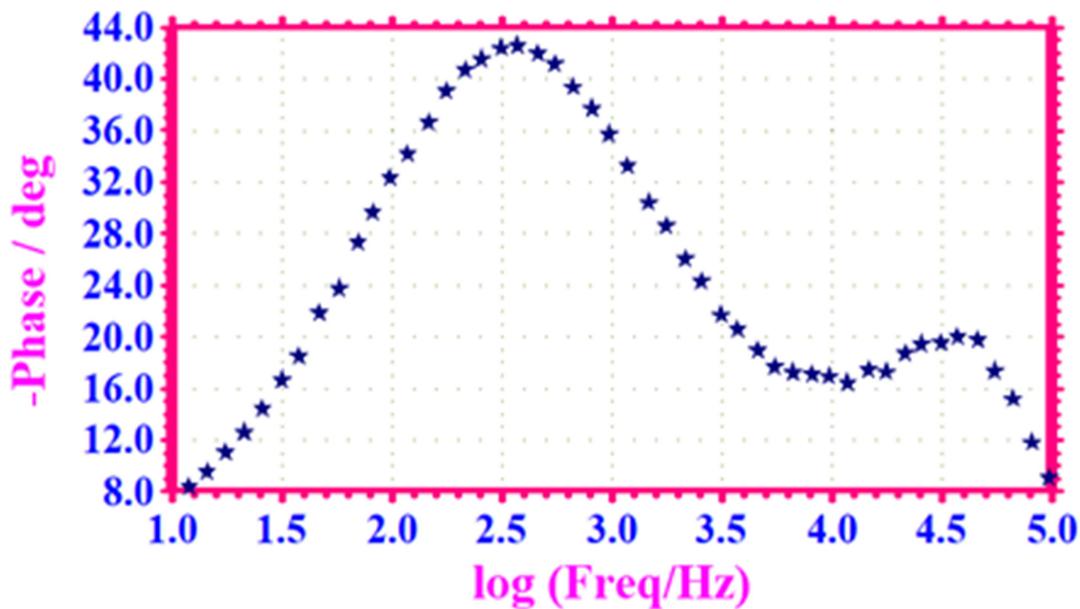

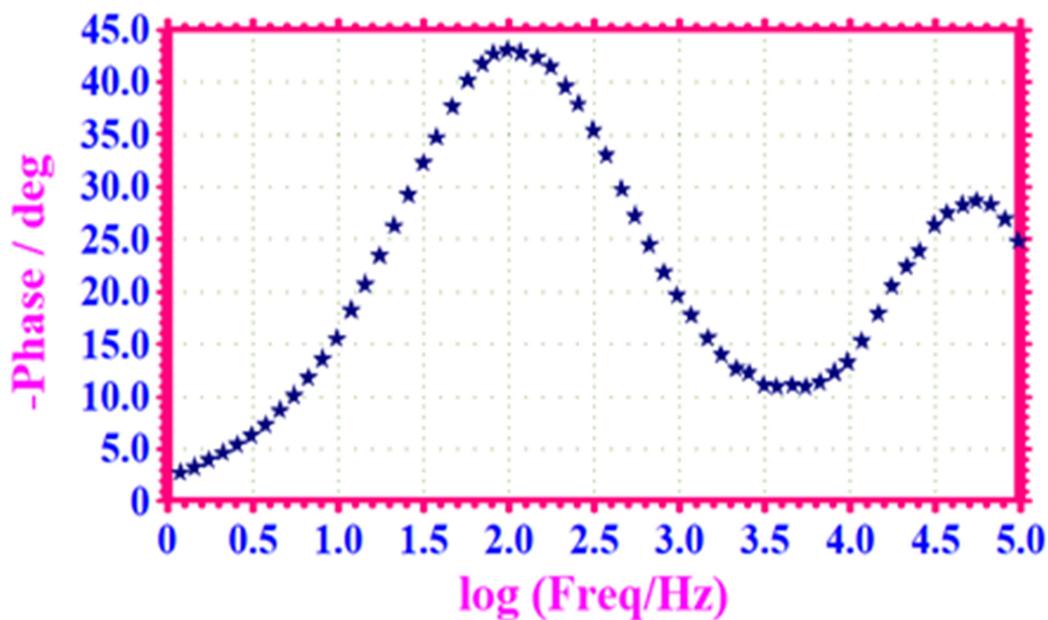**Figure 4a: AC impedance spectra of carbon steel immersed in 0.5M HCl (Phase angle)**

Figure 4b: AC impedance spectra of carbon steel immersed in 0.5M HCl with 10% aqueous leaves extract of OCLP (Phase angle)
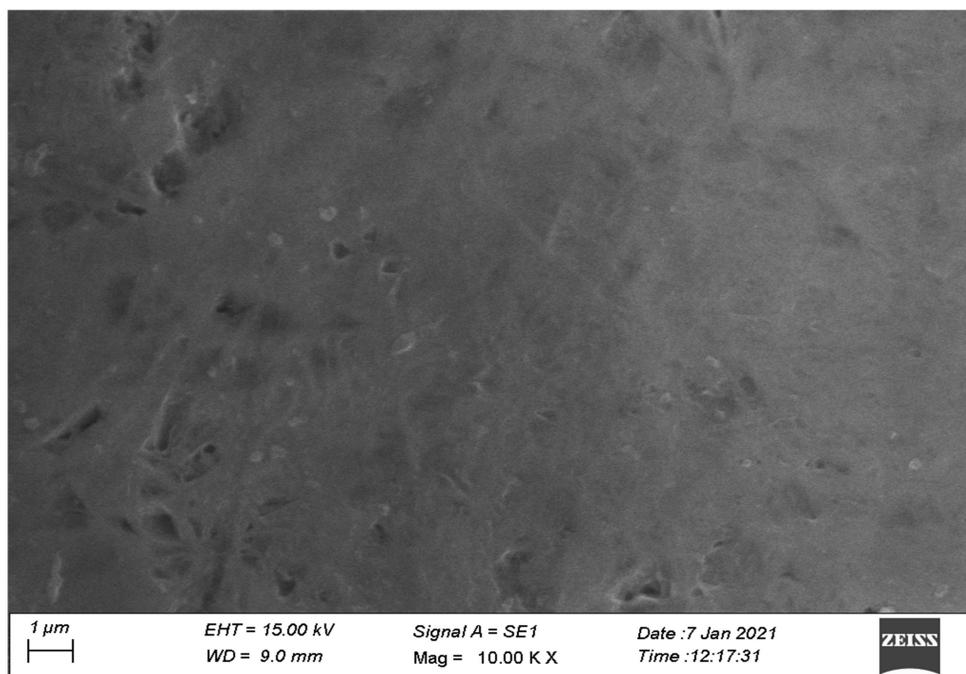

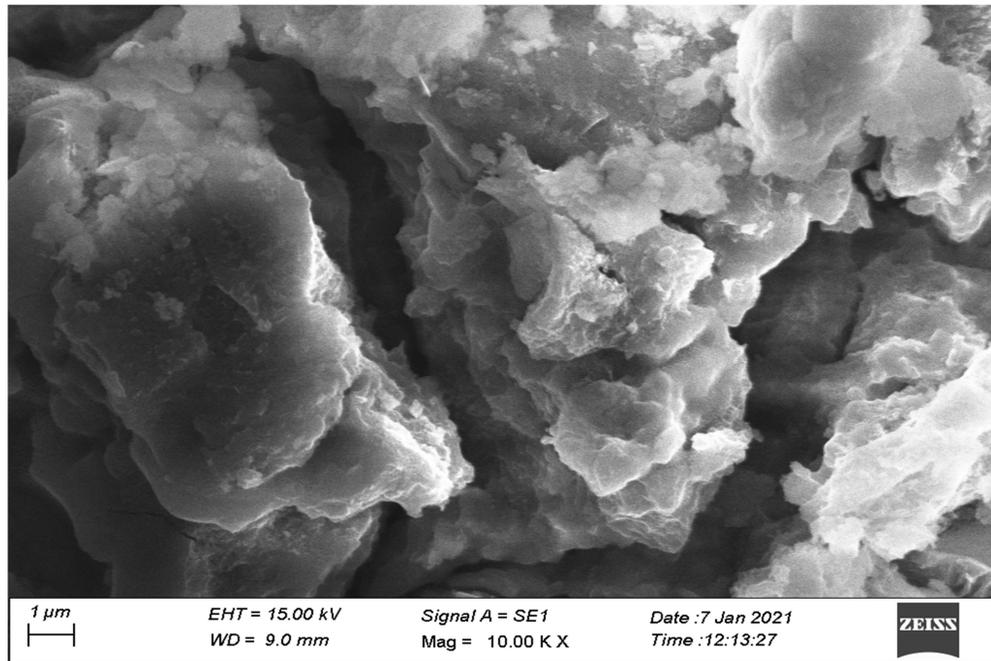**Figure 5a: SEM image of polished carbon steel specimen before immersion in 0.5M HCl (control)**

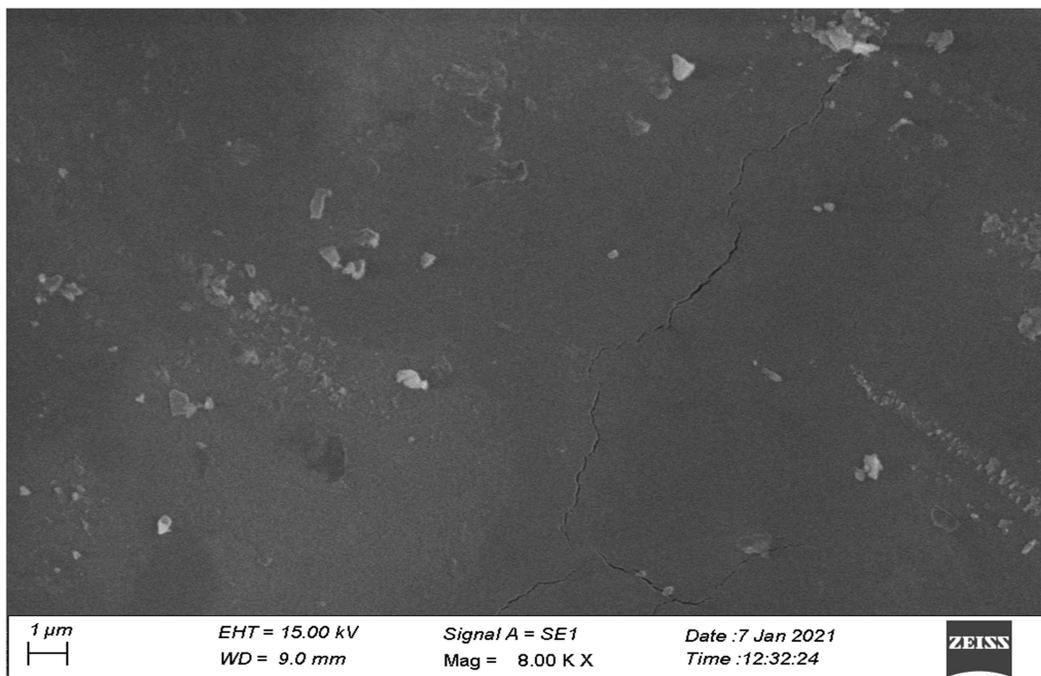**Figure 5b: SEM image of carbon steel specimen after immersion in 0.5M HCl (blank)**

Figure 5c: SEM image of polished carbon steel specimen after immersion in 0.5M HCl in the presence of 10% aqueous extract of OCLPL

DISCUSSION

Analysis of results of mass loss method

The corrosion rate was found to depend on the concentration of the inhibitor. Increasing the concentration of inhibitor increases the inhibition efficiency IE% which reached its maximum value at a concentration of 10 ml of aqueous leaves extract of *oxalis corniculata linn* plant. It is observed that aqueous leaves extract of *oxalis corniculata linn* plant offers 93.50% of inhibition efficiency. The inhibitory efficiency and corrosion rate rise as the content of aqueous leaves extract of oxalis corniculata linn plant increases [16]. This is due to an increase in surface coverage at greater inhibitor concentrations, which limits the dissolving of carbon steel by blocking their corrosion sites and thereby slowing the corrosion rate, with increasing efficiency as inhibitor concentrations rise. The presence of many phytochemical constituents in the plant leaves extracts is responsible for the inhibition of carbon steel corrosion. There may be reasons for the anti-corrosive actions of plant leaves extract and the same assumption has been already reported. This surveillance is in good agreement with the results reported by many researchers [17-19].

Analysis of results of potentiodynamic polarization study

It is observed **(Figure 1a)** that when carbon steel is immersed in 0.5M HCl, the corrosion potential is – 512 mV Vs SCE (Saturated Calomel Electrode). The LPR value is 118.4 Ohm/cm^2. The corrosion current is 1.771×10^{-6} A/cm^2. The corrosion potential is moved to the anodic side (- 441 mV / SCE) (Figure 6b) when 10 % of OCLPLE is added to the above corrosive environment. Due to the creation of a protective coating on the carbon steel surface, the corrosion potential is moved to the anodic side. By producing Fe2+-OCLPLE combination on the anodic sites of the carbon steel surface, this film limits the anodic reaction of carbon steel dissolution [20-23]. An increase in LPR and decrease in I_{corr} values are indications of the more corrosion-resistant nature of the inhibitor system.

Analysis of results of alternating current impedance spectra

When carbon steel is immersed in 0.5M HCl, R_t value is 12.94 ohm cm^2 and C_{dl} value is 1.2727×10^{-7} F cm^{-2}. R_t value increases from 12.94 to 33.05 ohm cm^2 when 10% of OCLPLE is added to 0.5M HCl blank system. The C_{dl} value is also decreased from 1.2727×10^{-7} to 4.9833×10^{-8} F cm^{-2}. The

impedance value [log (z/ohm)] increases from 1.1613 to 1.3006. Furthermore, the phase angle of the inhibitor system increases from 43.5^o to 44.7^o when compared to the blank system [24-27]. This recommends that a protective layer is formed on the surface of the carbon steel surface.

Surface analysis of carbon steel by SEM

The SEM images of smooth carbon steel surface in **Figure 5a** indicate the absence of any corrosion inhibitor formed on the carbon steel surface [28]. **Figure 5b** SEM image of rough carbon steel surface which indicates the highly corroded area. The figure 5c SEM image of carbon steel surface in presence of inhibitor is less corroded surface and smooth carbon steel surface which is due to the strong adsorption of the corrosion inhibitor on the carbon steel surface and suppress the corrosion process.

However, in the presence of an inhibitor (10 percent OCLPLE), the rate of corrosion is slowed, as seen by the reduction in corroded areas. Because of the creation of an insoluble compound on the surface of the carbon steel, it is almost corrosion-free. The smoothness of the carbon steel specimen is almost identical to that of the polished carbon steel surface [29]. In the presence of OCLPLE, the surface is covered by a thin

layer of inhibitor which effectively controls the dissolution of carbon steel immersed in 0.5M HCl with 10 % of aqueous extract of OCLPL. Thus it is revealed that the inhibitor has increased the efficiency of adsorption at the carbon steel/solution interface and thus the inhibitors tend to reduce metallic surface destruction.

CONCLUSION

In this present investigation undergoes the corrosion inhibition of carbon steel by using an aqueous leaves extract of *oxalis corniculata linn* plant to control the corrosion of carbon steel engrossed in 0.5M HCl. The following points were concluded based on the above-obtained results

➢ The aqueous leaves extract of *oxalis corniculata linn* plant act as a good corrosion inhibitor in controlling the corrosion of carbon steel is immersed in 0.5M HCl and shows good corrosion inhibition efficiency.

➢ Polarization study indicates that the systems function as anodic inhibitors controlling the anodic reaction predominantly.

➢ The mass loss technique shows the inhibition efficiency is 93.50%.

➢ Electrochemical impedance measurements indicate that an increase the charge transfer resistance

(R_t), decrease the double-layer capacitance (C_{dl}) and corrosion current (I_{corr}) values owing to the increased thickness of the adsorbed layer.

➢ SEM micrographs show the smoothness of carbon steel surfaces like polished carbon steel.

Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of this article.

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