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CORROSION INHIBITION EFFECT OF AN AQUEOUS EXTRACT OF *OXALIS ACETULOSA* PLANT LEAVES ON MILD STEEL IMMERSED IN 1M HYDROCHLORIC ACID

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ABSTRACT

Corrosion inhibition effect of an aqueous extract of *oxalis acetulosa* plant leaves on mild steel immersed in 1M hydrochloric acid. The corrosion inhibition efficiency and corrosion rates have been determined by weight loss method. The mechanistic aspects of corrosion inhibition have been studied by electrochemical studies such as potentiodynamic polarisation technique and electrochemical impedance spectroscopy (EIS). It is observed that as the concentration of the inhibitor increases the corrosion rate decreases and the inhibition efficiency increases. This is due to adsorption of the molecules of the active ingredients of the extract on the metal surface. A maximum inhibition efficiency of 89.60% is achieved by this inhibitor system. Potentiodynamic polarisation technique reveals that the inhibitor system functions as an anodic type of inhibitor, controlling anodic reaction preferably. It is observed that, in presence of inhibitor, linear polarization resistance (LPR) value increases from 405 ohm.cm^2 to 1889.5 ohm.cm^2. Corrosion current (I_{corr}) decreases from 1.022×10^{-4} A/cm^2 to 2.370×10^{-5} A/cm^2. Charge transfer resistance (R_t) value increases from 98.75 ohm.cm^2 to 432.2 ohm.cm^2. Double layer capacitance (C_{dl}) value

decreases from 1.6607×10^{-8} F cm^{-2} to 3.7945×10^{-9} F cm^{-2}. The impedance value [log (z/ohm)] increases from 0.350 to 0.510. The phase angle value increases from 12.5 to 28.0°. Electrochemical studies reveal that a protective film is formed on the metal surface. The surface morphology of the protective film has been studied by SEM and AFM. The outcome of the study can be used in pickling industry, wherein, hydrochloric acid is used to remove the rust on the mild steel surface.

Keywords*:* **Acidic solutions, AFM, Mild steel corrosion, oxalis acetulosa plant leaves, Scanning Electron Microscopy and Weight loss method**

INTRODUCTION

Mild steel finds applications in automobile body components, structural shapes, sheets, etc because of its properties such as strength, ductility, toughness, malleability, machinability and weldability. The use of hydrochloric acid as media in the study of corrosion of mild steel has become important because of its industrial applications such as acid pickling, industrial cleaning, acid descaling, oil-well acid in oil recovery and petrochemical processes **[1-3]**. Generally acid using materials undergo corrosion and it is inevitable. This corrosion induced in the material, besides loss in weight and cross section, can lead to hostile effects on the material properties. The refining of crude oil were carried out in a variety of corrosive conditions and in such, the corrosion of equipments are generally caused by a strong acid through attacking on equipment surface. Therefore, it is required to prevent or reduce it by using inhibitors or additives. Even

though various techniques like surface modifications, anodic and cathodic protections, and coating (painting) for the metal are available, the use of inhibitors in the medium is found to be one of the simple and cost-effective choices to protect metals against corrosion, particularly for a closed system **[4-6]**. Inhibitors are used in the industrial process to control metal dissolution especially in acid, neutral and base environment. Most of the efficient inhibitors used in industry are the organic compounds that possess at least one functional group, which is considered as the active center for the adsorption process. Several researchers have made an attempt to study the inhibition action of various organic compounds on the corrosion of aluminium, alloys, mild steel and composites in acids, alkaline and neutral media **[7-8]** The adsorption of inhibitor molecules on surface of carbon steel block the active sites of

carbon steel reduces the rate of corrosion. Use of organic compounds as corrosion inhibitors cause the environment pollution and harmful effects to human being. Literature survey has reported that aqueous extract of plant leaves could be used as corrosion inhibitors. They are called as green inhibitors which do not cause any environmental pollution and not harmful to human health **[9-10]**. Oil extract of plant materials has been used as inhibitor for corrosion resistance of mild steel (hull plate) in sea water by Dorothy et al. [11]. Mild steel has to be in contact with sea water which contains aggressive sodium chloride ions to the extent of 3.5%. This leads to severe corrosion problems. To mitigate this corrosion problem an oil coating containing extracts of natural products has been used. The corrosion protection nature of this oil coating has been evaluated by weight loss method which reveals that the oil coating offers 99% inhibition efficiency to mild steel immersed in natural sea water. The polarization study reveals that the coating acts as a barrier film controlling the anodic reaction predominantly. SEM images of various metal surfaces reveal that in the presence of sea water alone, pits are noticed on mild steel whereas in the presence of oil coating the surface appears to be smooth,

when immersed in sea water. When mild steel is immersed in sea water, the contact angle decreases when compared with polished metal. But for oil coated mild steel the contact angle increases, hydrophobicity increases and hence corrosion protection increases. It is suggested that this oil coating containing extracts of plant material may be coated on hull plates made of mild steel to protect them from severe corrosion due to the aggressive ions present in sea water [12]. Shahini et al. have used chamomile flower extract as a green corrosion inhibitor for mild steel in HCl solution. A 98% inhibition efficiency was obtained. The surface morphology of the protective film has been analysed by FE-SEM, EDAX, AFM, and contact angle examinations **[13]**. Alcoholic extract of a seaweed Sargassum Muticum has been used in controlling corrosion of mild steel in 0.5 N HCl by Jeslina et al **[14]**. The inhibitive effect of an alcoholic extract of a seaweed, namely, Sargassum Muticum, in controlling corrosion of mild steel in 0.5 N HCl has been evaluated by weight loss method (immersion period 1 day) and electrochemical techniques such as polarization study and AC impedance spectra. The protective film has been investigated by AFM and Vickers Hardness test. Weight loss method reveals that 500

ppm of inhibitor offers 99% inhibition efficiency to mild steel immersed in 0.5 N HCl. Fouda et al. have used Plant Doum (*Hyphaene thebaica* L.) as a safe corrosion inhibitor for carbon steel in a solution of HCl. Plant Doum (*Hyphaene thebaica* L.) extract was studied as a corrosion inhibitor for carbon steel (CS) dipped in 1 M hydrochloric acid using chemical and electrochemical tests. From chemical tests based on mass loss (ML), the inhibition efficiency increased with increasing the concentration of Doum (*Hyphaene thebaica* L.) extract (up to 150 ppm). The adsorption isotherm of Doum (*Hyphaene thebaica* L.) extract on CS surface obeys the Langmuir adsorption isotherm. The inhibition efficiency improves on raising the concentration of the Doum (*Hyphaene thebaica* L.) extract and decreases with an increase in the temperature of the medium. This extract may form a film which acts as a barrier decreasing the contact area between the CS surface and the HCl solution. Doum (*Hyphaene thebaica* L.) extract acts as mixed inhibitor in HCl solution without modifying the mechanism of hydrogen evolution. From the EIS examination, it was noticed that with an increase in the concentration of Doum (*Hyphaene thebaica* L.) extract, the double layer capacitance decreased, but the charge transfer resistance increased **[15]**.

The present study is to utilize the aqueous extract of *oxalis acetulosa* plant leaves as inhibitor to control the corrosion of mild steel immersed in 1M HCl. The effectiveness of inhibitor in terms of corrosion rate and inhibition efficiency has been evaluated by weight loss method. The mechanistic aspects of corrosion inhibition is determined by electrochemical studies such as AC impedance spectra and polarization studies. The protective film was formed over the surface of mild steel has been analyzed by scanning electron microscopy technique. The smoothness of mild steel when compared to polished mild steel, corroded mild steel (blank) and mild steel in inhibitor system have been characterized by Scanning Electron Microscopy (SEM). The roughness of carbon steel surface has been analyzed by Atomic Force Microscopy [AFM].

MATERIALS AND METHODS

Mild steel specimens

Carbon - 0.1 %, Sulphur - 0.026 %, Phosphorus - 0.06 %, Manganese - 0.4 % and the balance iron of dimensions 1.0 cm × 4.0 cm × 0.2 cm were polished to mirror finish and degreased with acetone and used for weight loss method. The corrosion environment (1M HCl) was prepared by

dilution of an analytical grade hydrochloric acid with double distilled water.

Preparation of inhibitor solutions

An aqueous extract of leaves of *oxalis acetulosa* plant was prepared by boiling 10 g of shade dried leaves with double distilled water. The suspended impurities were removed by filtration. The solution was made upto 100 ml and used as corrosion inhibitor.

Weight loss method

Mild steel specimens were immersed in 1M hydrochloric acid for 2 hours without and with different concentration (2, 4, 6, 8 and 10%) of inhibitor.

After the elapsed time, the specimens were taken out, washed, dried and weighed accurately.

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

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

Where W_i and W_o are the weight loss values in g in presence and absence of an inhibitor.

Electrochemical Techniques

In the present work corrosion resistance of mild steel immersed in various test solutions were measured by Polarization study and AC impedance spectra. Electrochemical measurements were performed in a CHI-

electrochemical work station with impedance model 660A.

Polarization study

Polarization studies were carried out in a three electrode cell assembly. A SCE was used as the reference electrode. Platinum was the counter electrode. Mild steel was the working electrode. From polarization study, corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes anodic = b_a, and cathodic = b_c ,and LPR (linear polarisation resistance) values were measured **[19-20]**.

AC Impedance spectra

The same instrument and experimental set-up used for polarization study was used to record AC impedance spectra also. A time interval of 5 to 10 min was given for the system to attain a steady state open circuit model. The real part (Z') and imaginary part (-Z'') of the cell impedance were measured in ohms at various frequencies. AC impedance spectra were recorded with initial E(V) = 0, high frequency ($1-10^5$ Hz), low frequency (1 Hz), amplitude (V) = 0.005 and quiet time (s) = 2. From Nyquist plot the values of charge transfer resistance (R_t) and the double layer capacitance (C_{dl}) values were calculated. From Bode plot the values of impedance and phase angle were calculated **[21]**.

Surface Examination Techniques

The mild steel specimens were immersed in blank, as well as inhibitor solutions, for a period of 2 hours. After 2 hours, the specimens were taken out and dried. The nature of the film formed on the surface of the mild steel specimens was analyzed by various analysis techniques such as SEM and AFM.

Scanning Electron Microscope (SEM)

Thus SEM was used to analyze the topography of the mild steel surface after corroding in presence and absence of the inhibitor [22]. The SEM images were recorded by the SEM instrument, JEOL MODEL JSM 6390.

Atomic Force Microscopy (AFM)

The surface morphology of mild steel surface in the absence and presence of inhibitor were recorded by Atomic Force Microscopy (AFM) using SPM Veeco di Innova connected with the software version V7.00 and the scan rate of 0.7Hz [23]. 2D images, 3D images and sectional analysis were recorded. The AFM parameters such as S_a, S_q, S_y and S_p were measured.

RESULTS

Weight loss studies

The present study an aqueous extract of *oxalis acetulosa* plant leaves has been used as inhibitor to control the corrosion of mild steel immersed in 1M HCl. The effectiveness of inhibitor in terms of corrosion rate and inhibition efficiency has been evaluated by weight loss method. The corrosion rates (CR) of mild steel immersed in a 1M HCl and also inhibition efficiencies (IE) in the absence and presence of the extract of the *oxalis acetulosa* inhibitor obtained by weight loss method are given in **Table 1**.

Analysis of potentiodynamic polarization study

Polarization study has been used to confirm the formation of protective film on the mild steel surface during corrosion inhibition process. If a protective film is formed on the mild steel surface, the linear polarization resistance value (LPR) increases and the corrosion current value (I_{corr}) decreases.

The potendiodynamic polarization curves of mild steel immersed in 1M hydrochloric acid in the absence and presence of inhibitor are shown in **Figure 1 (a, b).** The corrosion parameters are given in **Table 2**.

Analysis of AC impedance spectra

AC impedance spectra (electrochemical impedance spectra) have been used to confirm the formation of protective film on the mild steel surface. If a protective film is formed on the mild steel surface, charge transfer resistance (R_t) increases; double layer capacitance value (C_{dl}) decreases and the impedance log (z/ohm) value increases.

The AC impedance spectra of mild steel immersed in 1M hydrochloric acid in the absence and presence of inhibitor (10% of aqueous extract of *oxalis acetulosa* plant leaves) are shown in **Figures 2 and 3 (a, b)** and the corrosion parameters are given in **Table 3**.

SEM Analysis of mild steel surface

SEM provides a pictorial representation of the surface of mild steel. To understand the nature of the surface film in the absence and presence of inhibitors and extent of corrosion of mild steel, the SEM micrographs of the surface are examined.

The SEM images of mild steel specimens immersed in 1M HCl for two hours in the presence and absence of inhibitor system are shown in **Figures 4 (a, b and c). Figure 4a** shows the SEM image of polished mild steel surface. **Figure 4b** shows the SEM image of polished mild steel immersed in corrosive medium, namely 1M HCl for 2 hours. **Figure 4c** shows the SEM image of polished mild steel immersed in 1M HCl and inhibitor system for a period of 2 hours.

Analysis of Atomic force microscopy

The surface morphology protective films on mild steel have been examined with AFM. Atomic force Microscopy is a powerful tool for the gathering of roughness statistics from a variety of surfaces. Roughness in any surface is easily investigated by AFM studies. Atomic force microscopy provides direct insight view into the changes in the surface morphology that takes place at several hundred nanometers, when topographical changes take place between corrosion and the formation of protective film on the mild steel surface in the absence and presence of inhibitors.

This characterization contains three dimensional (3D) AFM morphologies and the AFM cross-sectional profile for polished mild steel surface, mild steel in 1M HCl (blank sample), mild steel surface with corrosion inhibitor immersed in 1M HCl are shown in **Figures 5a, 5b and 5c**.

AFM images analysis was performed to obtain the average surface roughness, S_a (the average deviation of all points roughness profile from a mean line over the evaluation length), root-mean-square surface roughness, S_q (the average of the measured height deviations taken within the evaluation length and measured from the mean line), S_y the maximum peak-to-valley (largest single peak-to-valley height in five adjoining sampling heights) and S_p maximum peak height (Maximum profile peak height indicates the point along the sampling length at which the curve is highest). The AFM parameters are given in **Table 4**.

Table 1: Inhibition efficiency of aqueous extract of *oxalis acetulosa* plant leaves in controlling corrosion of mild steel in 1M HCl at room temperature (303K)

Concentration of Inhibitor (%)	Corrosion rate (mmd)	Inhibition Efficiency (%)
Blank	265.70	-
2	124.80	53.00
4	103.40	61.10
6	78.50	70.50
8	53.50	79.90
10	25.00	89.60

Table 2: Corrosion parameter of mild steel immersed in 1M hydrochloric acid in the absence and presence of inhibitor system obtained by potentiodynamic polarization method

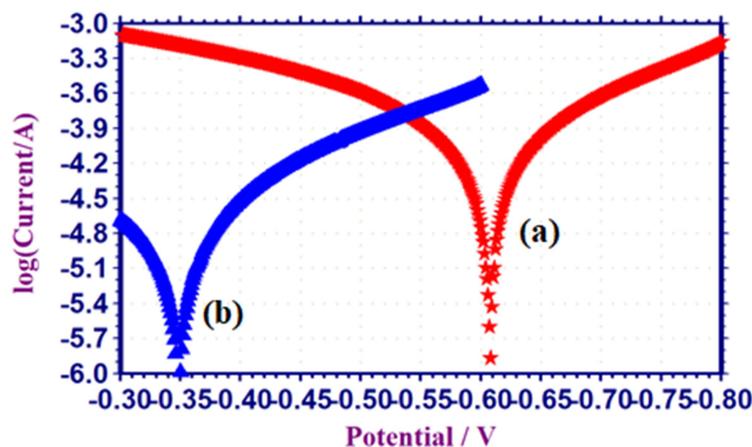| Concentration of the aqueous extract of OAPL (mL)	E_{corr} vs SCE (mV)	I_{corr} (A/cm^2)	b_a (mV/dec)	b_c (mV/dec)	LPR (ohm. cm^2)
0	- 608	1.022×10^{-4}	198	182	405
10	- 348	2.370×10^{-5}	285	161	1889.5

Figure 1: Potentiodynamic polarization curves of mild steel in 1M HCl
(a) in 1M HCl (blank)
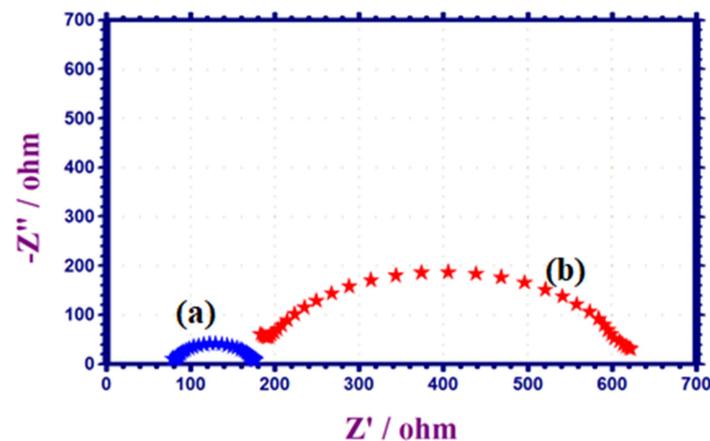**(b) in 1M HCl + inhibitor extract**

Figure 2: AC impedance spectra of mild steel immersed in 1M HCl in the absence and presence of aqueous extract of *oxalis acetulosa* plant leaves inhibitor (Nyquist plots)
(a) Mild steel in 1M HCl without inhibitor
(b) Mild steel in 1M HCl with 10% aqueous extract of *oxalis acetulosa* plant leaves.

Table 3: Electrochemical impedance parameters of mild steel immersed in the absence and presence of aqueous extract of *oxalis acetulosa* plant leaves

Concentration of the aqueous extract of BRPL (mL)	Nyquist plot		Impedance Log (z/ohm)	Phase angle (°)
	R_t, Ohm.cm^2	C_{dl} F/cm^2		
0	98.75	1.6607×10^{-8}	0.350	12.5
10	432.2	3.7945×10^{-9}	0.510	28.0
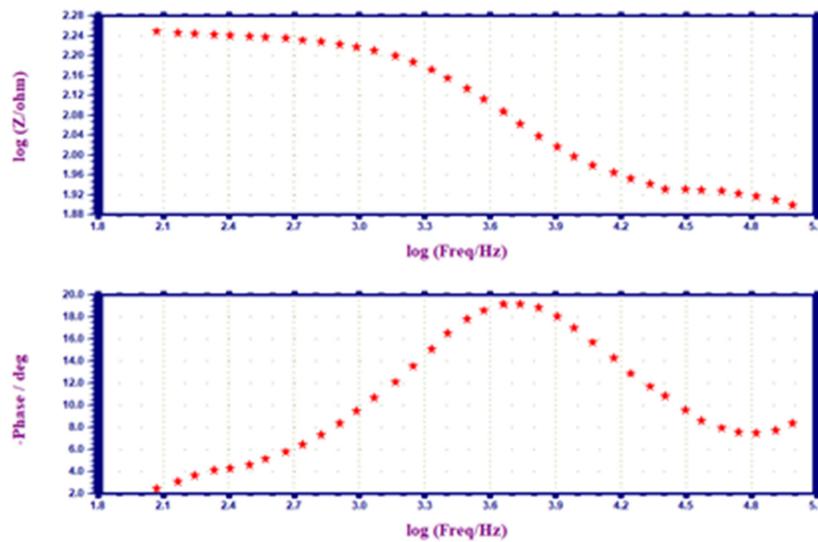
Figure 3a: AC impedance spectra of mild steel immersed in 1M HCl (Bode Plot)
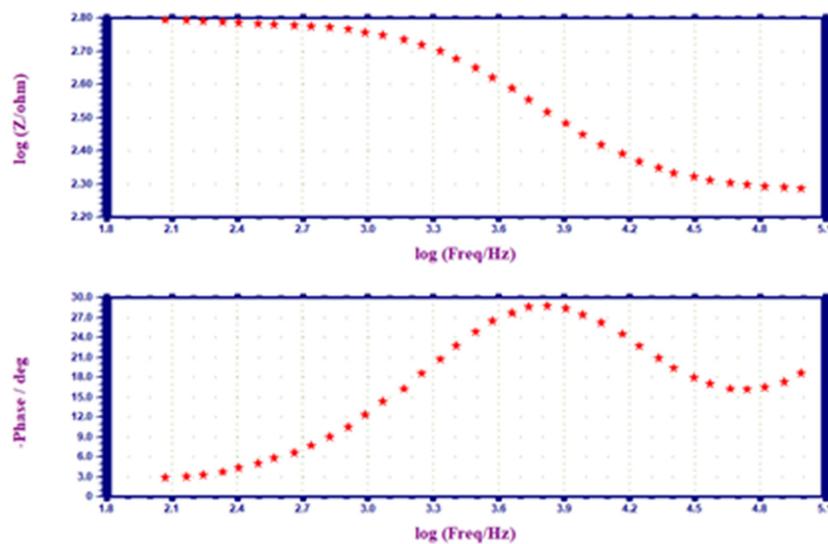

Figure 3b: AC impedance spectra of mild steel immersed in 1M HCl with 10% of an aqueous extract of OAPL (Bode Plot)
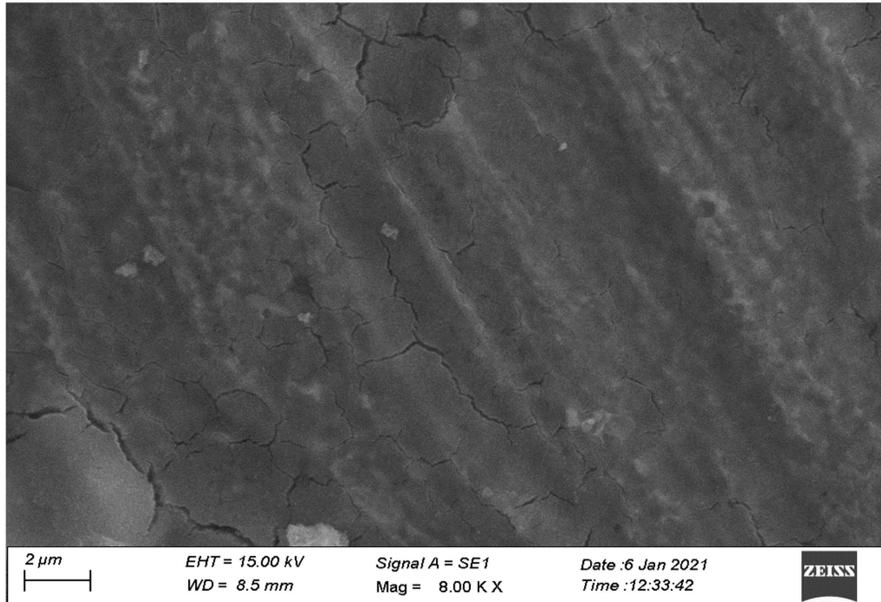
Figure 4a: SEM image of polished mild steel specimen before immersion in 1M HCl (control)
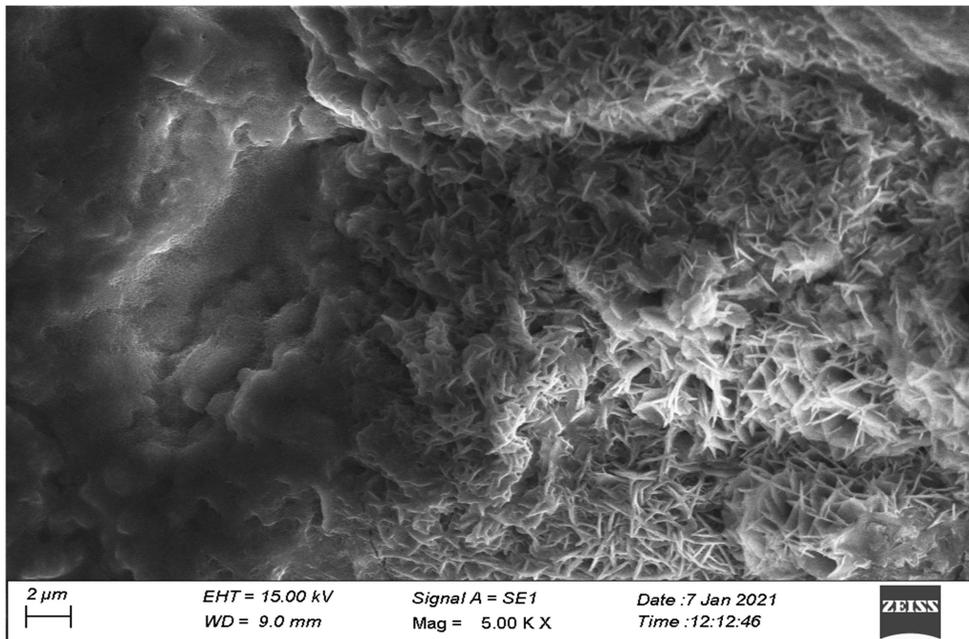

Figure 4b: SEM image of mild steel specimen after immersion in 1M HCl (blank)
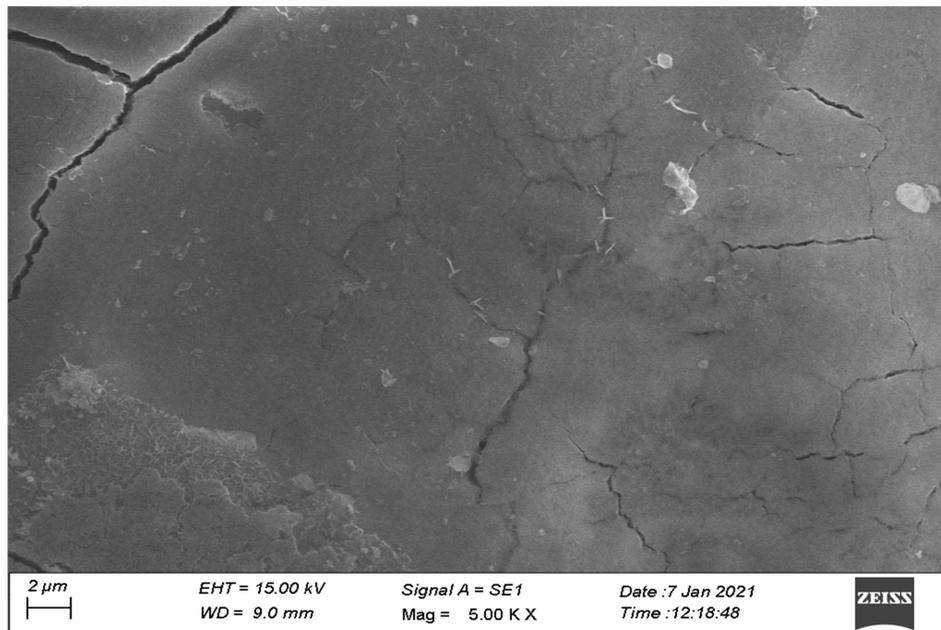
Figure 4c: SEM image of polished mild steel specimen after immersion in 1M HCl in the presence of 10% of an aqueous extract of OAPL

Table 4: AFM data for mild steel immersed in the presence and absence of inhibitor systems

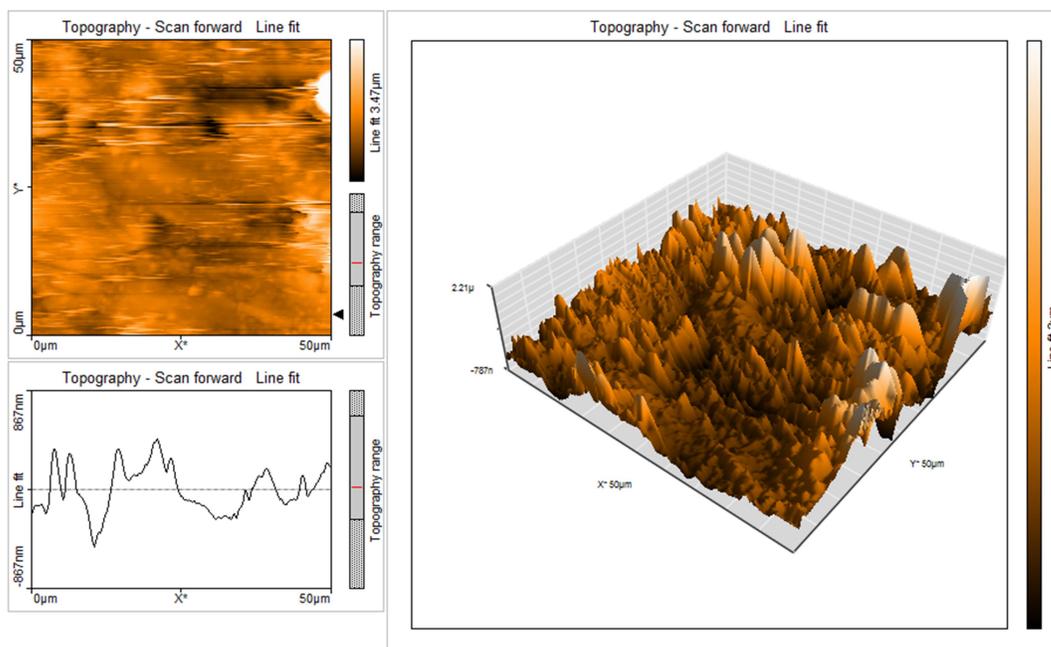| Samples	Sa nm	Sq nm	Sy nm	Sp nm
Mild steel surface	291.79	433.21	5778.90	3795.00
Mild steel surface immersed in 1M HCl	1182.60	1443.50	13046.0	6031.60
Mild steel surface immersed in 1M HCl + 10% of an aqueous extract of OAPL	770.93	998.05	11967.00	5015.10

Figure 5a: AFM cross sectional image of the polished mild steel surface (control)
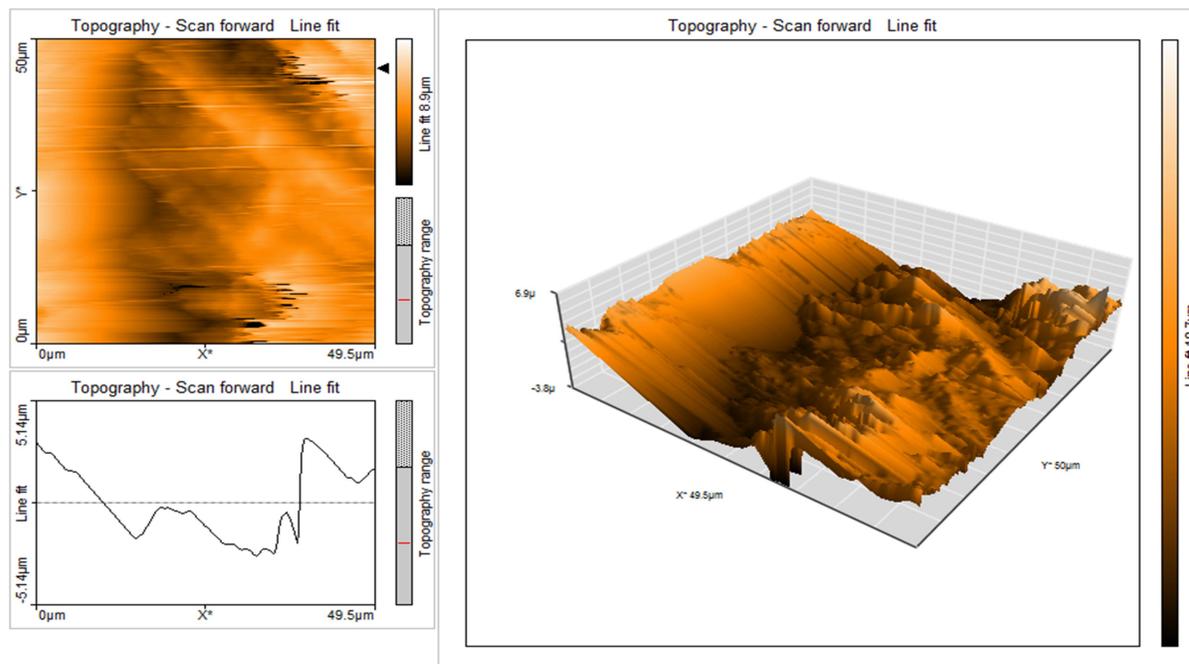
Figure 5b: AFM cross sectional image of the mild steel surface after immersion in 1M HCl (blank)
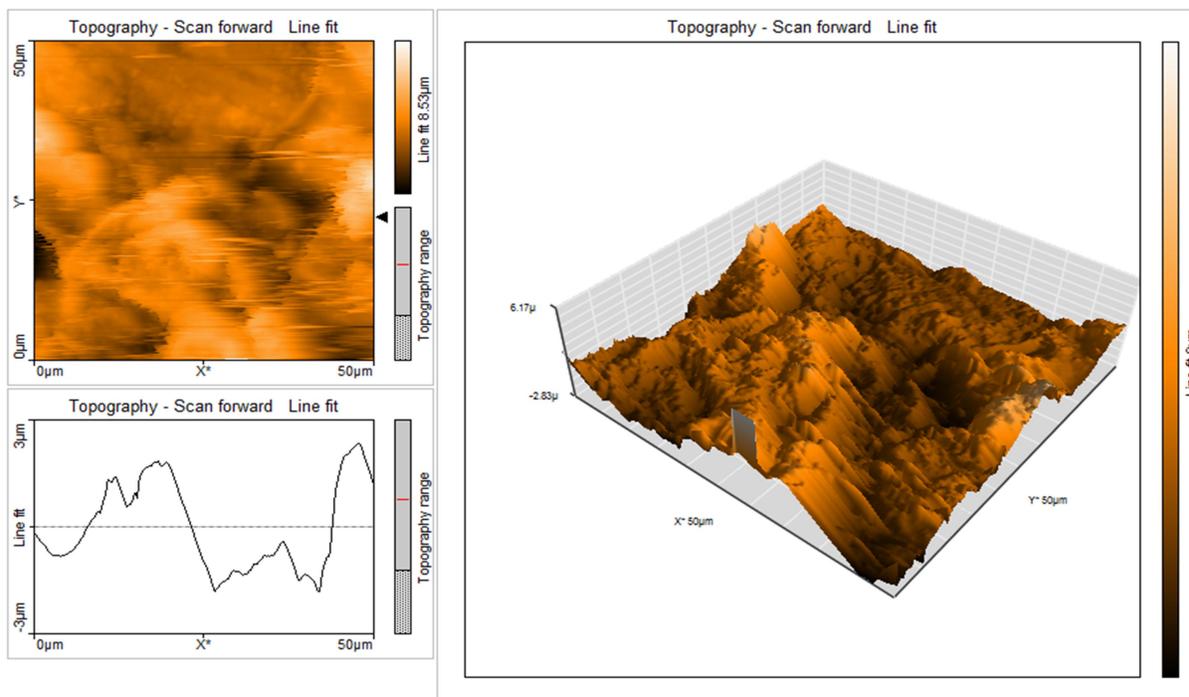

Figure 5c: AFM cross sectional image for the mild steel surface after immersion in 1M HCl with 10% of an aqueous leaves extract of OALP

DISCUSSION

Analysis of weight loss studies

It is observed that 10% of the extract of *oxalis acetulosa* offers 89.60 % of inhibition efficiency. It is observed from **Table 1** that as the concentration of the extract of *oxalis acetulosa* increases, the IE increases. This is due to an increase of surface coverage at higher concentration of the *oxalis acetulosa* which retards dissolution of mild steel. A protective film is formed on the metal surface. It consists of Fe^{2+} - active principles (present in the extract) complex. The possibility of interaction between the hetero atoms such as N and O present in the plant leaves extract and metal ion from the metal surface can be attributed for higher inhibition efficiencies. The presence of many phytochemical constituents in the plant extracts are responsible for the inhibition of mild steel corrosion There may be the reasons for the anti-corrosive actions of plant extracts. This surveillance is in good agreement with the results reported by many researchers [24-25].

Analysis of results of potentiodynamic polarization study

When mild steel was immersed in 1M hydrochloric acid the corrosion potential was -608 mV vs SCE. When 10% of aqueous extract of *oxalis acetulosa* plant leaves was added to the above system, the corrosion potential was shifted to the anodic side -348 mV vs SCE. This indicates that the protective film is formed on the anodic sites of the mild steel surface. This film controls the anodic reaction of mild steel dissolution by forming Fe^{2+} - inhibitor complex on the anodic sites of the mild steel surface [26-29].

Further, the LPR value increases from 405 $ohm.cm^2$ to 1889.5 $ohm.cm^2$, the corrosion current decreases from 1.022×10^{-4} to 2.370×10^{-5} (A/cm^2). Thus polarization study confirms the formation of a protective film on the mild steel surface.

Analysis of results of AC impedance spectra

It is observed that when the inhibitor (10% of aqueous extract of *oxalis acetulosa* plant leaves) is added to the above positive system, the charge transfer resistance (R_t) increases from 98.75 $\Omega. cm^2$ to 432.2 $\Omega.cm^2$ and the C_{dl} value decreases from 1.6607×10^{-8} F cm^{-2} to 3.7945×10^{-9} F cm^{-2}. The impedance value [log (z/ohm)] increases from 0.350 to 0.510. Further, the phase angle value increases from 12.5 to 28.0^{o}. These results lead to the conclusion that a protective film is formed on the mild steel surface [30].

Analysis of SEM results

The SEM micrographs of polished mild steel surface (control) in **Figure 4a** shows the

smooth surface of the mild steel. This shows the absence of any corrosion products or inhibitor complex formed on the mild steel surface **[31].**

The SEM micrograph of mild steel surface immersed in 1M HCl **(Figure 4b)** shows the roughness of the mild steel surface which indicates the highly corroded surface of mild steel in 1M HCl. However, **Figure 4c** indicates that in the presence of inhibitor (10% of OAPL) the rate of corrosion is suppressed, as can be seen from the decrease of corroded areas. The mild steel surface is almost free from corrosion due to the formation of insoluble complex on the surface of the mild steel. In the presence of OAPL, the surface is covered by a thin layer of inhibitors which effectively controls the dissolution of mild steel **[32].**

 Analysis of the results of Atomic force microscopy

 It is observed that average surface roughness for mild steel in corrosive medium is very high. In presence of inhibitor, this value decreases. This value is lower than that of the corrosive medium (blank) but higher than that of the polished mild steel surface. This is due to the fact that, in presence of inhibitor, a protective film is formed on the mild steel surface. This film is found to be smooth. Similar is the case with, other three parameters namely root mean square roughness, maximum peak to valley height and maximum peak height **[33-36].**

CONCLUSION

In this study the aqueous leaves extract of OALP has been used as a corrosion inhibitor to prevent the corrosion of mild steel engrossed in 1M HCl. The present study leads to the following conclusion

➤ The aqueous leaves extract of OALP inhibitor shows good corrosion inhibition efficiency in controlling the corrosion of mild steel immersed in 1M HCl.

➤ Polarization study shows that the effective aqueous leaves extract of OALP systems function as anodic inhibitor controlling the anodic reaction predominantly.

➤ The weight loss technique shows the inhibition efficiency is 89.60%.

➤ Electrochemical 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 adsorbed layer.

- ➢ SEM micrographs show the smoothness of mild steel surface.
- ➢ The AFM microscopes confirm the smoothness of mild steel surface.

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