ANTI-CORROSIVE PROPERTIES OF *DATURA STRAMONIUM* IN ZINC METAL IMMERSED IN HYDROCHLORIC ACID

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Abstract

The anti-corrosive properties effect of zinc metal is 1N hydrochloric acid has been evaluated at room temperature by using conventional mass loss method. The corrosion rate and inhibition efficiency has been determined from mass loss method. The results of mass-loss show that 93.45% IE, in controlling the corrosion of zinc metal contains 1N HCl and 10% of *Datura stramonium* plant leaves as corrosion inhibitor. This is owing to at the higher concentration of inhibitor solution which prevents the active site of a zinc metal and a protective film is formed over the zinc metal surface. The rate of corrosion is decreased with increasing addition of additive such as green inhibitor, probably due to progressive adsorption of the phytochemical constituents present in the plant leaves on the zinc metal surface. Electrochemical studies have been used to observe the mechanistic aspects of corrosion and formation of protective film over the zinc metal surface. It is also recognized by surface analysis technique like FTIR and scanning electron microscopy. Smoothness and roughness of zinc metal surface has been observed and compared to blank and inhibitor system by SEM. The AFM microscopes confirm the roughness of zinc metal surface when compared to polished, blank and inhibitor system.

Keywords: *Anti-c*corrosion, AFM, DSPL, FTIR, Mass loss study and Scanning Electron Microscopy.

Introduction

Corrosion of metals results in the deterioration of the surface of metallic structure or metal alloy in the course of their chemical, electrochemical or biochemical interaction with the surrounding [1-3]. Zinc metal is one the most important alloys and can often be utilized as a construction frame material, incredibly high strength and structural steel beam. When it undergoes corrosion immersed in acids, alkalis, and sodium chloride solution. The use of well water and surface water in cooling system, storage reservoirs, concrete moulding purposes and water transport pipelines for injection systems and exhibit corrosion problems. When sea water contact with mild steel surface, it affects the mild steel and lead formation of corrosion products. It can be avoided by using inhibitor. The corrosive inhibitor is a significant method for defending materials against corrosion [4-6]. . Its effects of corrosion on the reliable safe and more efficient use of metal structures are often more severe than the simple loss of weight of metal. Mostly water and acid using materials undergoes corrosion and it is unavoidable one. Henceforth, it should be controlled it by spending corrosion inhibitors or corrosion additives. Corrosion inhibitor is a chemical substance and it is used to reduce the rate of corrosion when it is added to the environment may in acid, neutral and base medium. Plant extracts containing phytochemical constituents such as electron donating groups or polar functional groups, hetero atoms's like oxygen, nitrogen and sulphur atoms, aromatic rings with π- electrons are extensively employed as effective corrosion inhibitors in controlling the corrosion of metal surface immersed in corrosive medium. The corrosion resistors will be interacted on the metal surface either chemically or physically forming a protective film on the metal surface [7 - 9]. Scientist made an attempt to recognize the inhibitive effect of various plant extracts on the corrosion of metals such as aluminium, mild steel, zinc, copper, alloys, carbon steel and composites in acidic, basic and neutral environments [10- 13]. Scientists are involved to develop the non-toxic and easily available natural inhibitors. The naturally available plant extracts are considered as one of the rich source of environmentally acceptable and bio degradable corrosion inhibitors [14– 17]. The main objective of the present research work is to assess the effectiveness of aqueous extract of *Datura stramonium* plant leaves as inhibitor to control the corrosion of zinc metal dunked in 1N HCl solution. The performance of inhibitor in terms of corrosion rate and inhibition efficiency parameters has been found by mass loss method. The barrier film is formed on the surface of mild steel is confirmed by electrochemical studies such as AC impedance spectra and potentiodynamic polarization studies. The aggressive film was formed on the surface of mild steel has been examined by Fourier Transform Infra-red spectroscopy technique. The scanning electron microscopy has been used to analyze the nature of zinc metal surface. The roughness of zinc metal surface has been evaluated by AFM.

Materials and Methods

Corrosion inhibition of zinc metal in 1N hydrochloric acid solution by aqueous extract of *Datura stramonium* plant leaves has been examined.

Preparation of zinc metal specimens

Lead 1.03%, cadmium 0.04%, iron 0.001% and reminder being zinc were polished and used for mass loss measurements.

Preparation of stock solutions

DD water was used required in the preparation of solutions. The required strength of the aqueous extract of *Datura stramonium* plant leaves stock solution was prepared by dissolving the *Datura stramonium* plant leaves in minimum amount of water and making up to the desired volume with double distilled water.

Mass loss method

Mass loss measurements were performed for 3 hours by submersing the zinc metal specimens in 1N HCl solution in the absence and presence of different concentration (2, 4, 6, 8, 10 ml) of aqueous extract of *Datura stramonium* plant leaves inhibitor.
After elapsed time, the specimen was cleaned, dried and weighed accurately.

The effectiveness of inhibitor (IE %) was determined by the below relation [18]

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

Where W_i and W_O are the weight loss values in g in presence and absence of an aqueous extract of Datura stramonium plant leaves inhibitor.

Polarization study

Three electrode cell assembly was used in CHI- electrochemical work station with impedance model 660A. It was used for electrochemical measurements. The reference electrode is SCE. Platinum was the counter electrode. Zinc metal 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].

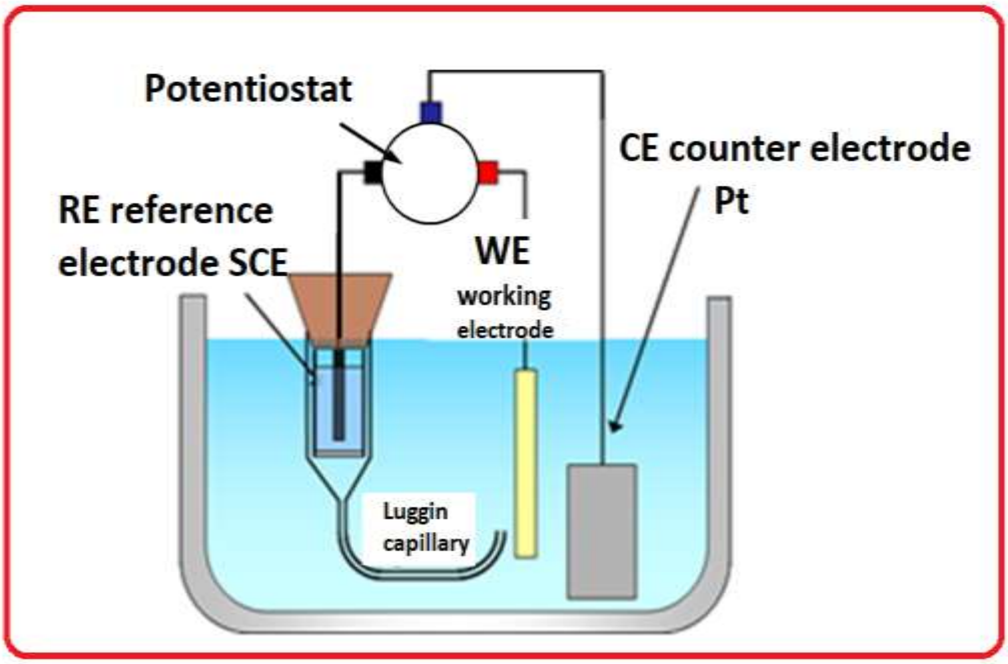


Figure 1. Three - electrode cell assembly

Alternating Current impedance measurements

An electrochemical workstation impedance analyzer CHI- electrochemical impedance model 660A. The cell setup was identical to that used to test polarization. The device was given a time interval of 5 to 10 minutes to reach a steady-state open circuit potential. An AC potential of 10 mV was then superimposed over this steady-state potential. For different frequencies, the AC frequency was varied from 100 kHz to 100 MHz, and the actual (z) and imaginary (z") sections of the cell impedance were calculated in ohms. The C_{dl} (double layer capacitance) and R_t (charge transfer resistance) values were determined. The following relationship was used to measure C_{dl} values.

$$C_{dl} = \frac{1}{2 \times 3.14 \times R_t \times f_{max}}$$

Surface characterization techniques

The zinc metal specimens were immersed in blank as well as extract of *Datura stramonium* plant leaves inhibitor solutions, for 3 hours. After one day, the specimens were taken out and dried. The nature of the surface protective film is covered on the surface of the zinc metal specimens was analyzed by different analysis techniques.

Surface analysis by FTIR spectra

FTIR spectra were recorded in a Perkin–Elmer 1600 spectrophotometer. The layer was removed, mixed thoroughly with KBr made into pellets and the FTIR spectra were recorded. After immersion period of one day in various environments, the specimens were taken out of the test solutions and dried. The film formed on the surface was scratched carefully and it was thoroughly mixed so as to make it uniform throughout [20].

Scanning Electron Microscopic studies (SEM)

SEM has been used to study the topography of the zinc metal surface after corroding absence and with presence of the inhibitor. SEM will be helpful to observe the difference in the nature of zinc metal surface before and after the metal surface is in direct contact with the corrodent solution and to notice the effect of the addition of the inhibitor [21]. The SEM image was taken by the SEM instrument, JEOL MODEL JSM 6390.

Atomic Force Microscopy (AFM)

Surface morphology was also characterized by using an atomic force microscope. After the inhibition test, the zinc metal specimens were placed in vacuum desiccators, mounted on sample holder under the objective of the atomic force microscope and the 3D -images were taken from the 100 × magnified surface through operating program on computer. The surface of zinc metal specimens after immersed in 1N HCl solution in the absence and presence of aqueous leaves extract of *Datura stramonium* plant for three hours were evaluated by atomic force microscopy analysis [22]. From the AFM cross sectional 3D – image, the line and surface roughness parameters of the specimens such as R_a, R_q and peak to valley value were obtained for the zinc metal after immersion in 1N HCl in the absence and presence of the inhibitor.

Results and Discussion

Analysis of mass loss method

Mass loss measurements were performed in 1N HCl solution in the absence and with presence of diverse concentrations of an aqueous extract of *Datura stramonium* plant leaves inhibitor. The inhibition efficiencies (IE) and corrosion rates (CR) of zinc metal is immersed in 1N HCl has been calculated in the absence and presence of aqueous extract of *Datura stramonium* plant leaves inhibitor by mass loss method. The inhibition efficiency and the corrosion rate values are given in table - 1.

Table 1. Corrosion rates (CR) and inhibition efficiency (IE %) were calculated by mass loss measurements for zinc metal is immersed in 1N HCl solution without and with various strength of aqueous extract of *Datura stramonium* plant leaves.

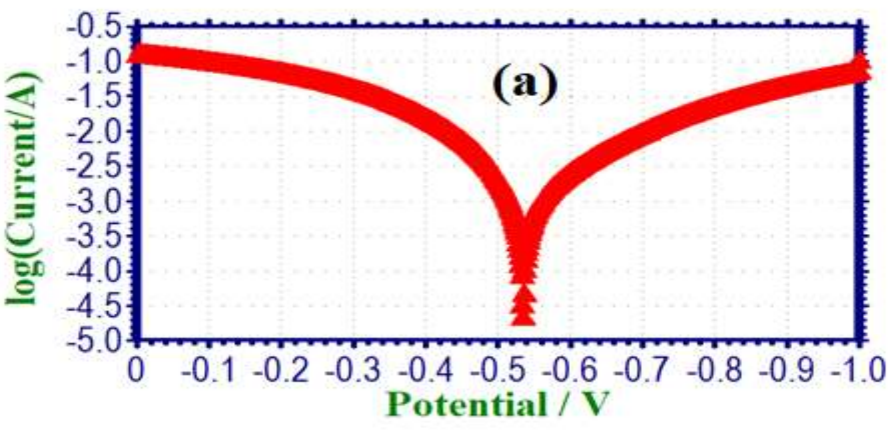
- Inhibitor System: Aqueous extract of *Datura stramonium* plant leaves
- Immersion period: 2 hours

Concentration of aqueous extract of DSPL inhibitor (%)	Corrosion rate (mdd)	Inhibition Efficiency (%)
blank	216.95	-
2	172.10	38.12
4	162.25	50.89
6	96.14	69.75
8	69.47	81.56
10	21.18	93.45

Increasing the strength of plant extract inhibitor, the inhibition efficiency (IE %) which achieved its maximum value at concentration of 10 ml of aqueous extract of *Datura stramonium* plant leaves. It is well-known that aqueous extract of *Datura stramonium* plant leaves offers 93.45% of inhibition efficiency. As the concentration of aqueous extract of *Datura stramonium* plant leaves increases, the corrosion rate decreases and inhibition efficiency increases [16]. This is due to an increase of zinc metal surface coverage. The maximum concentration of the inhibitor which control the dissolution of the zinc metal by securing the corroding sites and hence decreasing the corrosion rate, with increasing efficiency as their concentrations increase. The phytochemical constituents present in the plant leaves extracts is responsible for the corrosion resistance of zinc metal. This surveillance is in good agreement with the results described by other researchers [23-27].

Results of potentiodynamic polarization study

Polarization study is helpful to illustrate the protective film formation on the zinc metal surface. It permits the quick valuation of the action of inhibitor, stability of surface film and also the inhibition efficiency of corrosion inhibitors. If a protective film is covered on the zinc metal surface, the linear polarization resistance values (LPR) increases and the corrosion current value (I_{corr}) decreases. The potentiodynamic polarization curves of zinc metal immersed in 1N HCl solution in the absence and presence of plant extract inhibitor is shown in figure 2. The corrosion parameters namely corrosion potential (E_{corr}) Tafel slopes, b_c and b_a, linear polarization resistance (LPR) and corrosion current (I_{corr}) are given in Table 2.



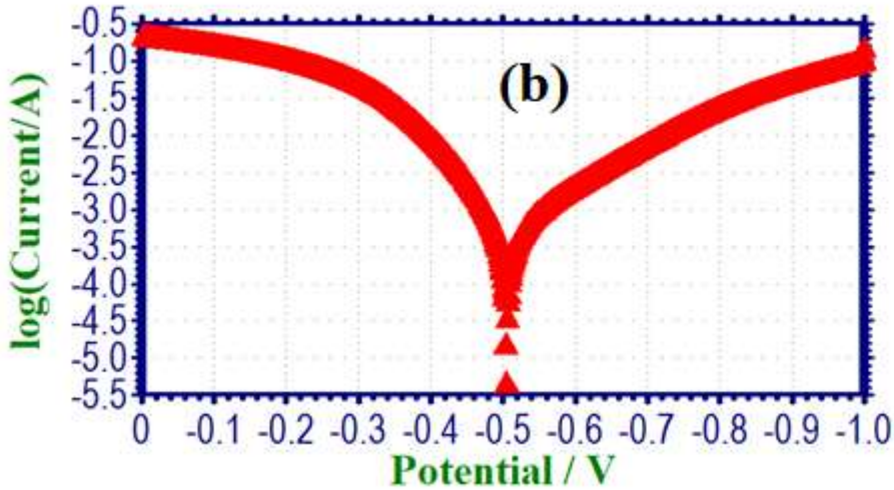


Figure 2. Potentiodynamic polarization curves for corrosion of zinc metal in 1N HCl solution absence and presence of DSPLE inhibitor
(a) Zinc metal in 1N HCl solution (blank)
(b) Zinc metal in 1N HCl with 10% aqueous extract of DSPL

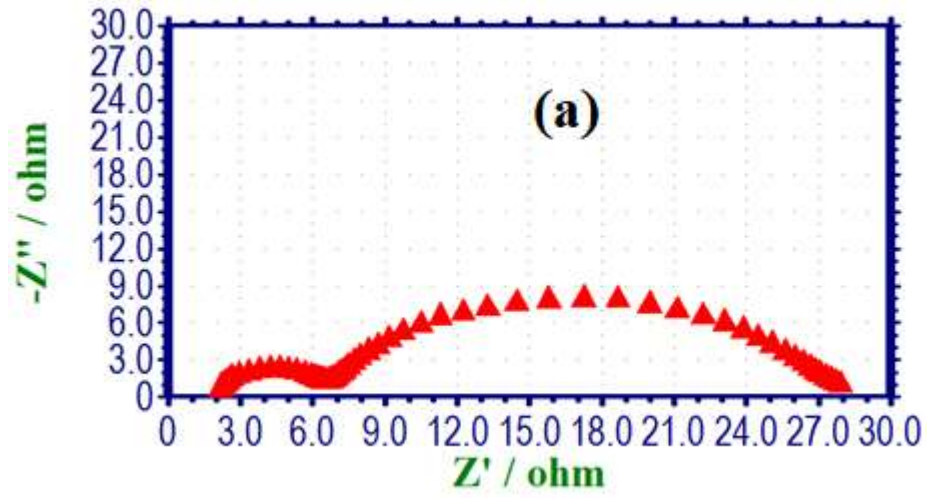
Table 2. Potentiodynamic Polarization parameters for the corrosion of zinc metal in 1N HCl solution for the aqueous extract of DSPL system

Concentration of the aqueous leaves extract of DSPLE (%V/V)	E_{corr} mV/SCE	Tafel slope		I_{corr} A / cm^2	LPR Ω/cm^2
		ba, mV/dec	bc, mV/dec		
blank	- 530	129	156	1.326×10^{-3}	23.9
10	- 508	091	163	5.258×10^{-4}	43.5

It is noticed that from Figure 2a when mild steel is engrossed in 1N HCl solution, the corrosion potential is – 530 mV Vs SCE (Saturated Calomel Electrode). The LPR value is 23.9 Ohm/cm^2. The corrosion current is 1.326×10^{-3} A/cm^2. The corrosion potential is shifted to the anodic side (- 508 mV / SCE) (Figure 2b) when 10 % of DSPLE is added to the above corrosive environment. This specifies that the corrosion potential is moved to the anodic side due to the formation of defensive layer on the zinc metal surface. This layer controls the anodic reaction of zinc metal dissolution by forming Zn^{2+}-DSPLE complex on the anodic sites of the zinc metal surface [28-30]. Decrease in I_{corr} and increase in LPR values are indications of more corrosion resistant nature of inhibitor system.

Results of impedance spectra

Electrochemical impedance spectra is supportive to endorse the protective film formation on the zinc metal surface. If a protective film is formed on the zinc metal surface, charge transfer resistance (R_t) increases; double layer capacitance value (C_{dl}) decreases and the impedance log (z/ohm) value increases. Electrochemical impedance spectra of zinc metal immersed in 1N HCl solution in the absence and presence of DSPLE inhibitor system are shown in figure. 3 (Nyquist plots) and figure 4 (Bode plots). The AC impedance parameters, namely, charge transfer resistance (R_t) and double layer capacitance (C_{dl}) are given in Table 3.



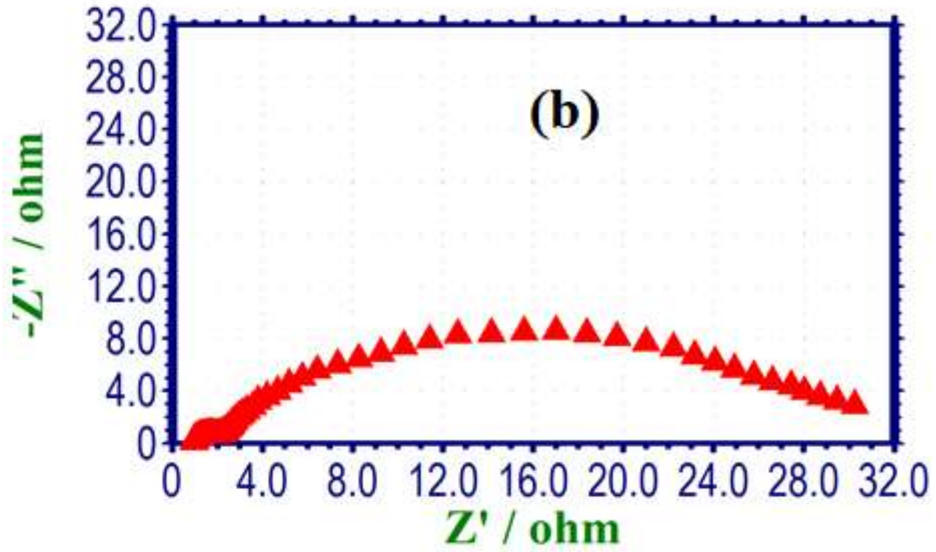


Figure 3. AC impedance spectra of zinc metal immersed in 1N HCl solution in the absence and presence of DSPLE inhibitor (Nyquist plots)
(a) Zinc metal in 1N HCl solution without inhibitor
(b) Zinc metal in 1N HCl solution with 10% aqueous extract of DSP leaves.

Table 3. Electrochemical impedance parameters from Nyquist plots for the corrosion of zinc metal for aqueous extract of DSP leaves in 1N HCl solution

Concentration of the aqueous extract of BCPL (%v/v)	Nyquist plot		Impedance Log (z/ohm)	Phase angle (degree)
	R_t, Ω/cm^2	C_{dl} F/cm^2		
blank	24.23	4.3285×10^{-4}	1.3	31.5
10	26.14	3.4893×10^{-5}	1.5	40.5

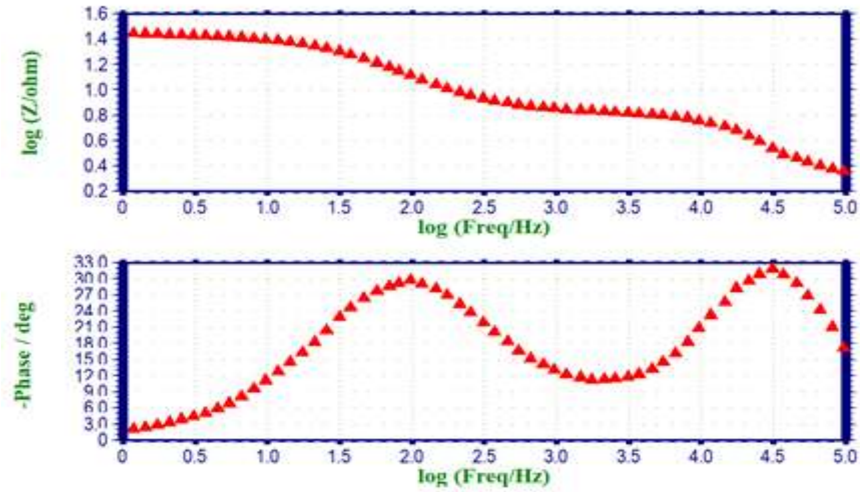


Figure 3a. AC impedance spectra of zinc metal immersed in 1N HCl solution (Bode Plot).

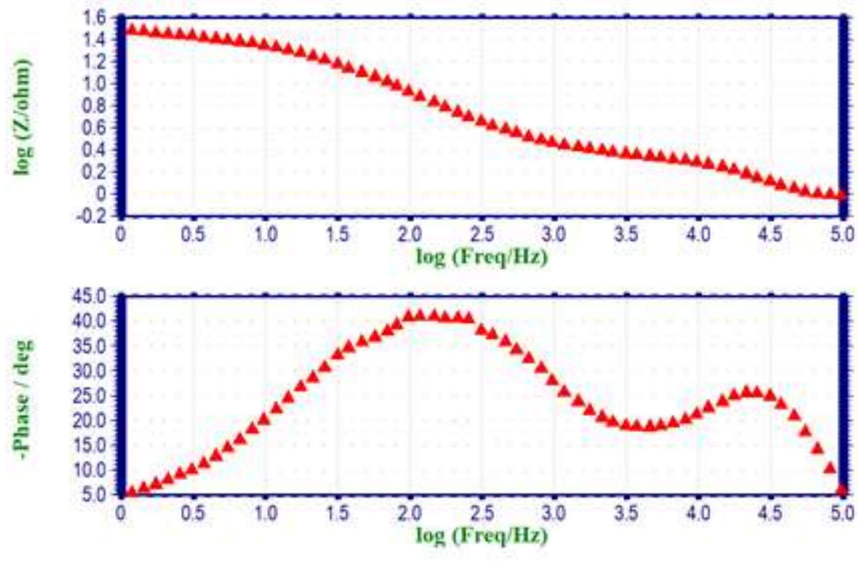


Figure 3b. AC impedance spectra of zinc metal immersed in 1N HCl solution with 10% aqueous extract of DSP leaves (Bode Plot).

It is observed that the zinc metal dipped in sea water solution, R_t value is 24.23 ohm cm^2 and C_{dl} value is 4.3285×10^{-4} F cm^{-2}. R_t value increases from 24.23 to 26.14 ohm cm^2 when 10% of DSPLE is added to sea water solution. The C_{dl} value is also decreases from 4.3285×10^{-4} to 3.4893×10^{-5} F cm^{-2}. The impedance value [log (z/ohm)] increases from 1.3 to 1.5. Furthermore, the phase angle of inhibitor system increases from 31.5^o to 40.5^o when compare to the blank system [31-34]. This suggests that an aggressive film is covered on the zinc metal surface.

Results of FTIR spectra

FTIR analysis helps to identify the absorption bands for the functional groups and the alignment of inhibitor molecules on the zinc metal surface. Many of the researchers have found that FTIR studies are a major tool that can be used to predict the nature of bonding of the inhibitor on the zinc metal surface. The absorption bands of the functional groups present in corresponding systems are given in Table -4. FTIR spectrum of aqueous extract of *Datura stramonium* is shown in Figure 4a. The C-H stretching frequency appears at 2920.13 cm^{-1}[35]. The C=N stretching frequency appears at 1680.20 cm^{-1}. The S-H stretching frequency appears at 2600.76 cm^{-1}. The peak due to C-C appears at 1010.53 cm^{-1}. The C-N frequency appears at 1590.36 cm^{-1} [36].

A Protective thin film is formed on the surface of the zinc metal immersed in 1N HCl with 10 ml of aqueous extract of *Datura stramonium* is shown in Figure 4b. A shift of the C-H stretching from 2920.13 to 3410.52 cm^{-1} indicates that the molecular adsorption [37 -39]. The shift in frequency from 2600.76 to 2720.53 cm^{-1} are noticed for S-H group. The shift in frequency from 1680.20 to 1710.84 cm^{-1} are noticed for C=N group. The C-C frequency is shifted from 1010.53 cm^{-1} to 1020.22 cm^{-1}. The peaks at 1590.02 cm^{-1} for C-N group is vanished. The band 560.94 cm^{-1} considerably originate mainly from Zn-complex [40]**.** All the above bands clearly indicate the formation of a complex on the zinc metal surface.

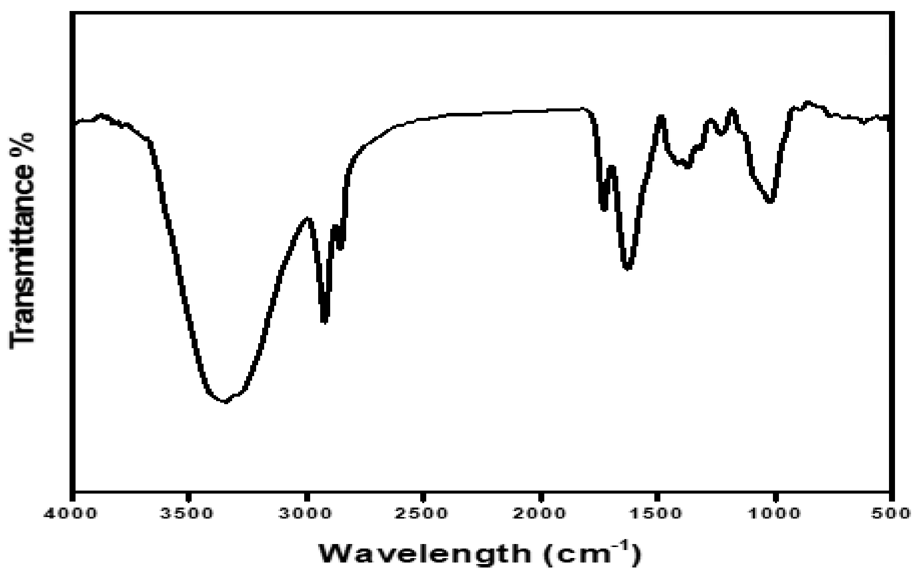


Figure – 4a: FT-IR spectrum of aqueous extract of *Datura stramonium*

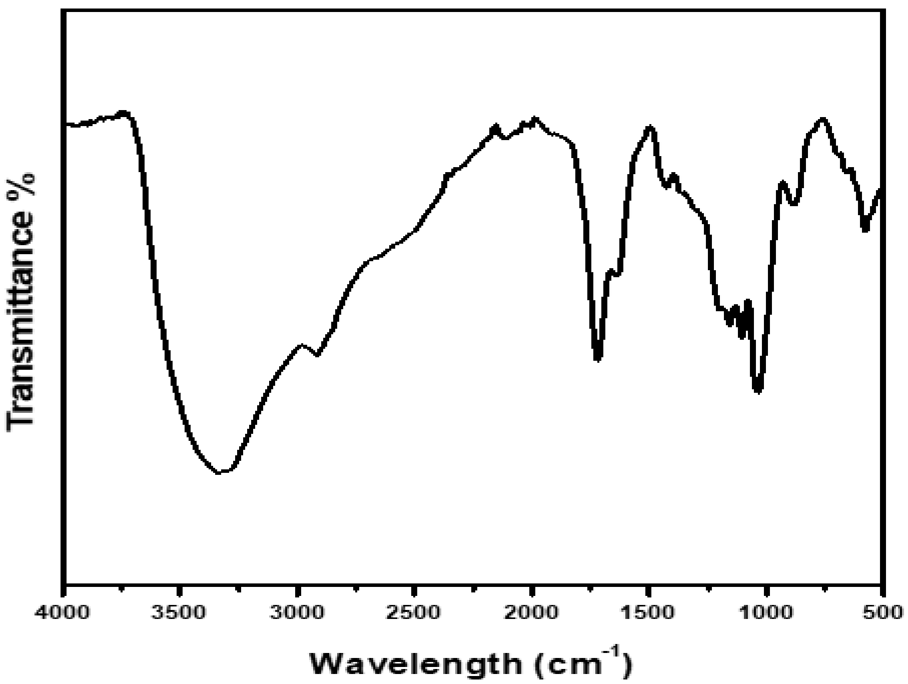


Figure –4b: FT-IR spectrum of scratched film from the zinc metal surface after immersion in 1N HCl with 10 ml of aqueous extract of *Datura stramonium*

Table – 4: FT-IR spectral data for the aqueous extract of *Datura stramonium* inhibitor and the scratched film from zinc metal surface after immersion in 1N HCl with 10 ml of DSPL inhibitor system.

IR bands of DSPLE inhibitor	IR Bands of film from zinc metal surface	Frequency assignment to functional groups
2920.13	3410.52	C-H stretching
1680.20	1710.84	C=N stretching
1010.53	1020.22	C-C stretching
2600.76	2720.53	SH
1590.36	-	C-N
-	560.94	Y-Fe_20_3

SEM Analysis of zinc metal surface

Scanning electron microscopy provides a pictorial representation of the zinc metal surface. To understand the nature of the film formed in the absence and presence of DSPL inhibitor on the zinc metal surface and the extent of corrosion of zinc metal, the SEM micrographs of the surface are examined [41-43]. The SEM images of zinc metal specimen immersed in 1N HCl for three hours in the absence and presence of DSPL inhibitor system are shown in Fig.5 (a, b & c) respectively.

The SEM micrographs of polished zinc metal surface (control) in Fig.5a shows the smooth surface of the zinc metal. This shows the absence of any corrosion products / rough surface or inhibitor complex formed on the zinc metal surface.

The SEM micrograph of zinc metal surface immersed in 1N HCl (Fig.5b) show the roughness of the zinc metal surface which indicates the highly corroded area of zinc metal immersed in 1N HCl medium**.**

However, in Figure.5c indicate that in the presence of inhibitor (10 ml of DSPLE) the rate of corrosion is suppressed, as can be seen from the decrease of corroded areas. The zinc metal surface almost free from corrosion due to the formation of insoluble complex on the surface of the zinc metal. The zinc metal specimen's smoothness show almost equal to the polished zinc metal surface [44]. In the presence of DSPLE, the surface is covered by a thin layer of inhibitor which effectively controls the dissolution of zinc metal immersed in 1N HCl with 10 ml of DSPL. Thus it is revealed that the inhibitor has increased the efficiency of adsorption at the zinc metal/solution interface and thus the inhibitors tend to reduce metallic surface destruction.

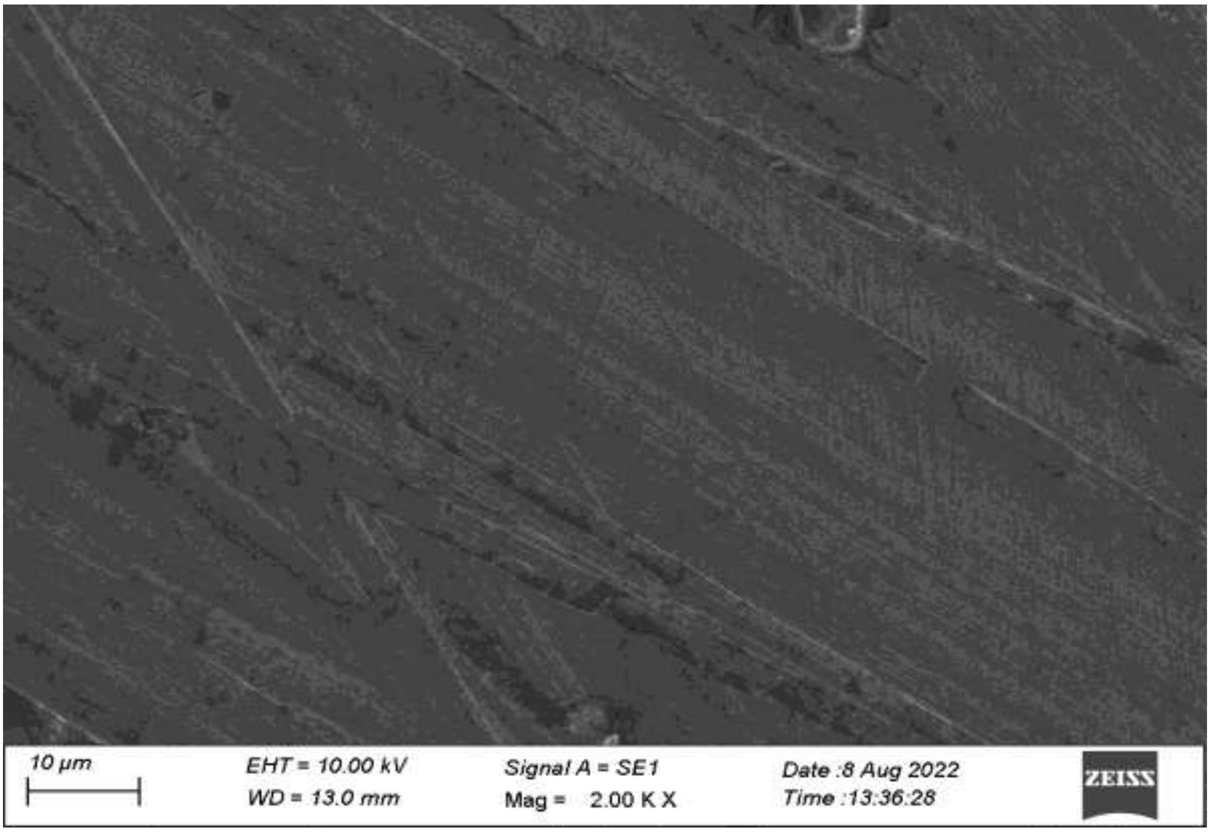


Figure - 5a: SEM image of polished zinc metal specimen before immersion in 1N HCl (control)

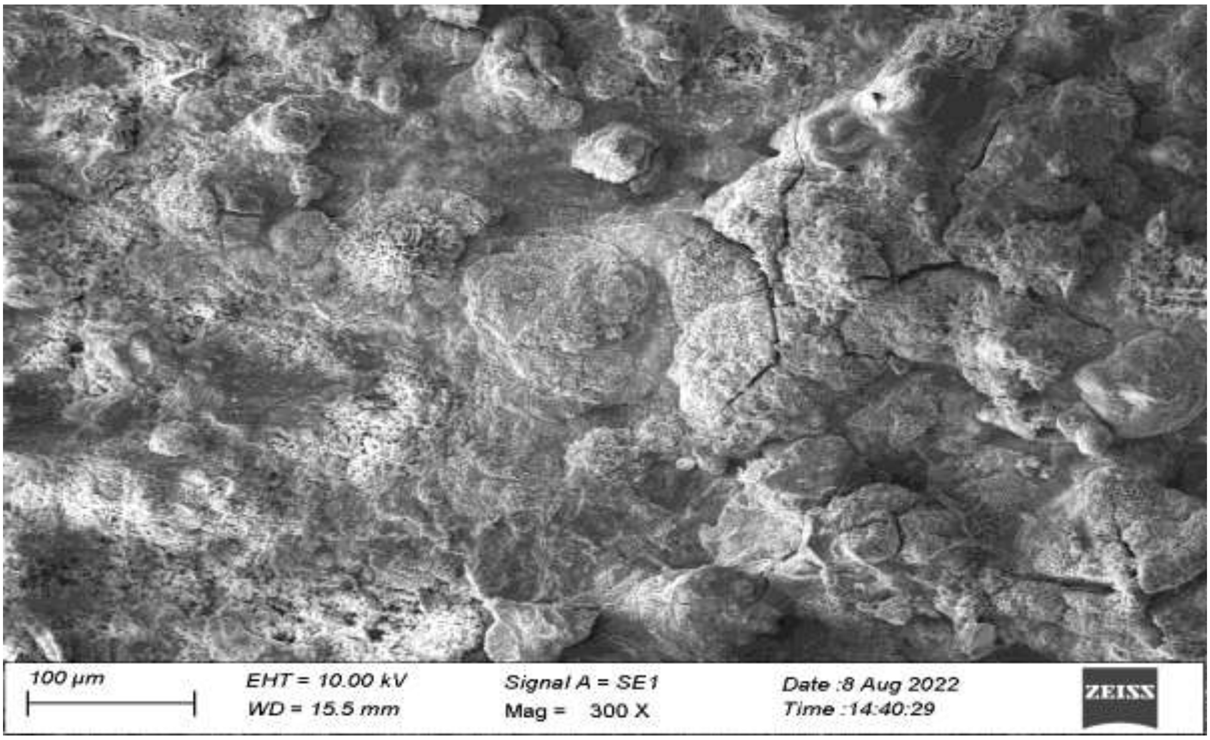


Figure - 5b: SEM image of zinc metal specimen after immersion in 1N HCl (blank)

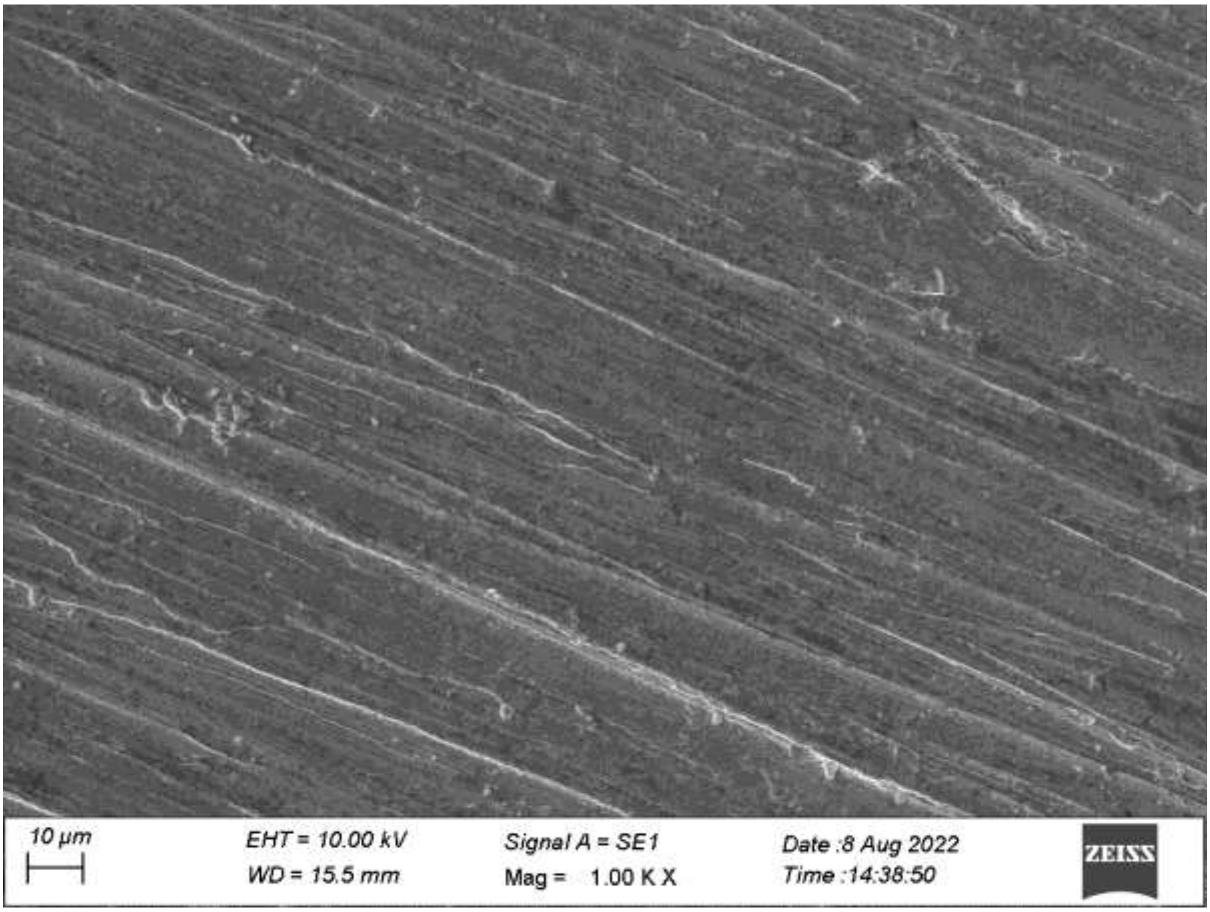


Figure - 5c: SEM image of polished zinc metal specimen after immersion in 1N HCl in the presence of 10 ml of DSPL.

Results of Atomic force microscopy

The surface morphology of protective films on zinc metal have been examined with AFM. Atomic force microscopy is a powerful tool for the gathering of roughness statistics from a variety of zinc metal 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 zinc metal surface in the absence and presence of inhibitors respectively [46].

This characterization contains three dimensional (3D) AFM morphologies and the AFM cross-sectional profile for polished zinc metal surface, zinc metal in 1N HCl (blank sample), mild steel surface with corrosion inhibitor immersed in 1N HCl are shown in Figures 6a, 6b and 6c.

AFM images analysis was performed to obtain the area roughness of 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 5.

Table - 5. AFM data for zinc metal immersed in the absence and presence of inhibitor systems (Area roughness)

Samples	S_a nm	S_q nm	S_y nm	S_p nm
Zinc metal surface	361.05	515.06	4225	2485.4
Zinc metal surface immersed in 1N HCl	955.93	1089.4	9942.6	5536.8
Zinc metal surface immersed in 1N HCl + 10 ml of DSPLE	479.81	621.28	4948.8	2984.7

It is observed that average surface roughness for zinc metal 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 zinc metal surface. This is due to the fact that, in presence of inhibitor, a protective film is formed on the zinc metal 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 [47].

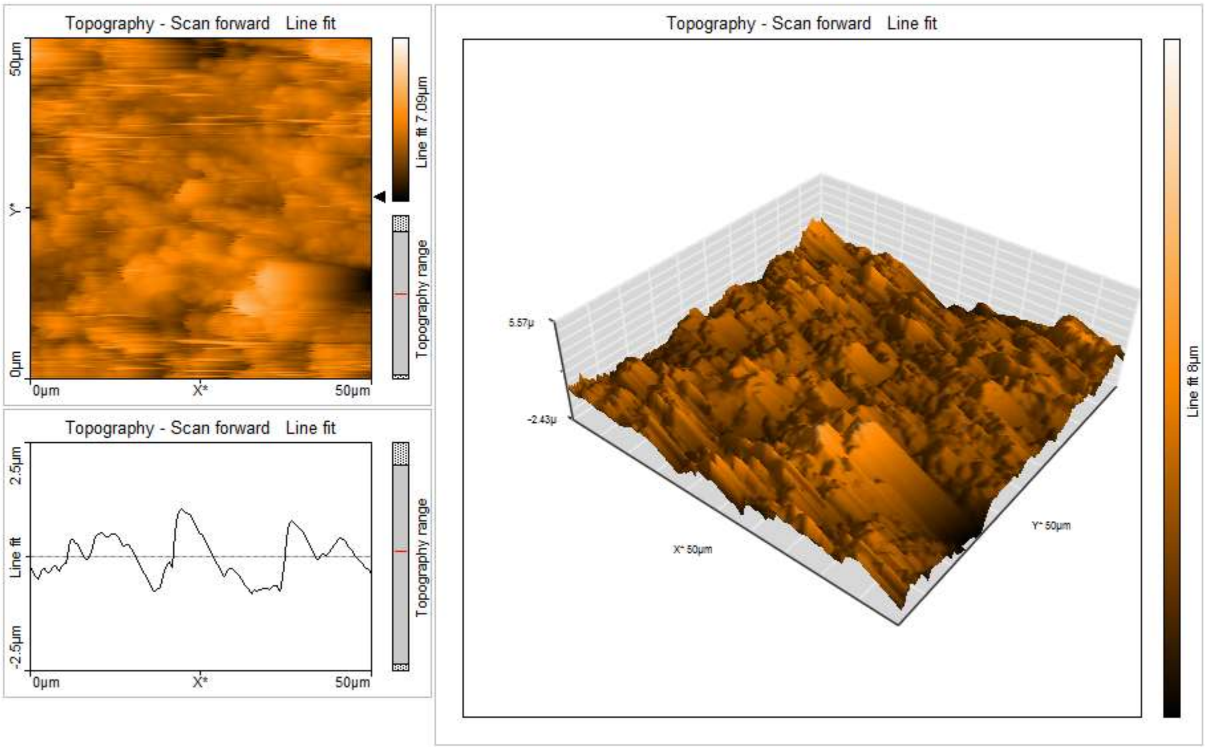


Figure - 6a: AFM cross sectional image of the polished zinc metal surface (control)

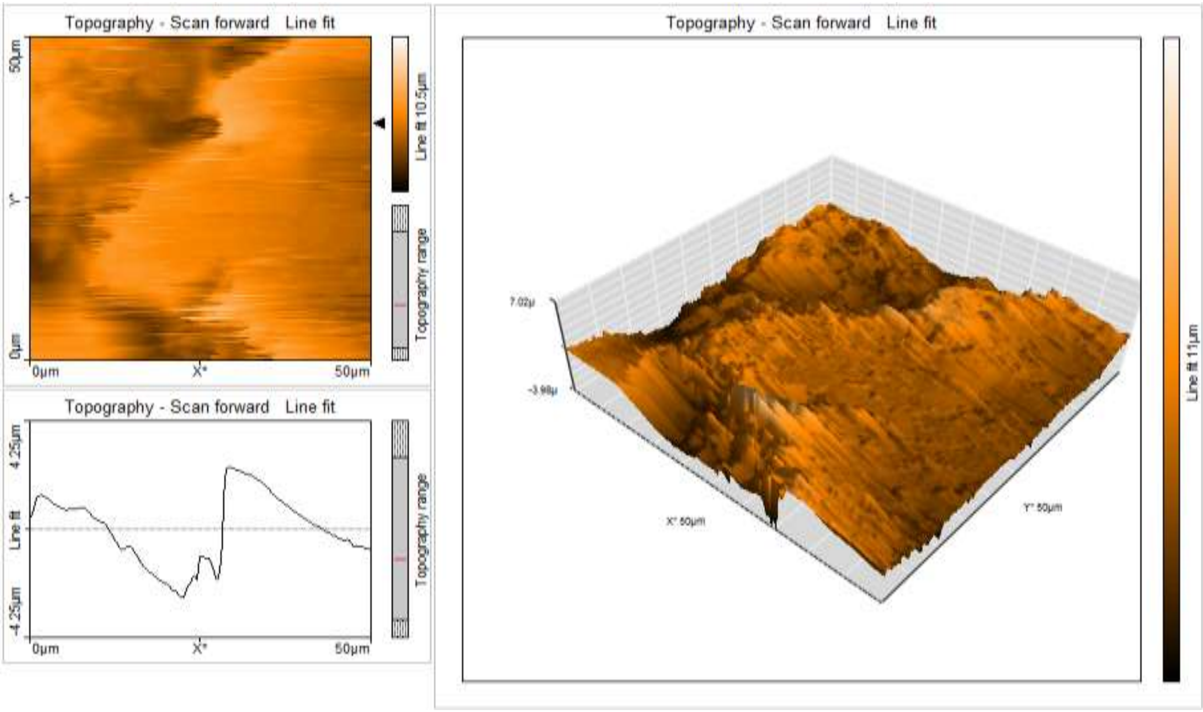


Figure - 6b: AFM cross sectional image of the zinc metal surface after immersion in 1N HCl (blank)

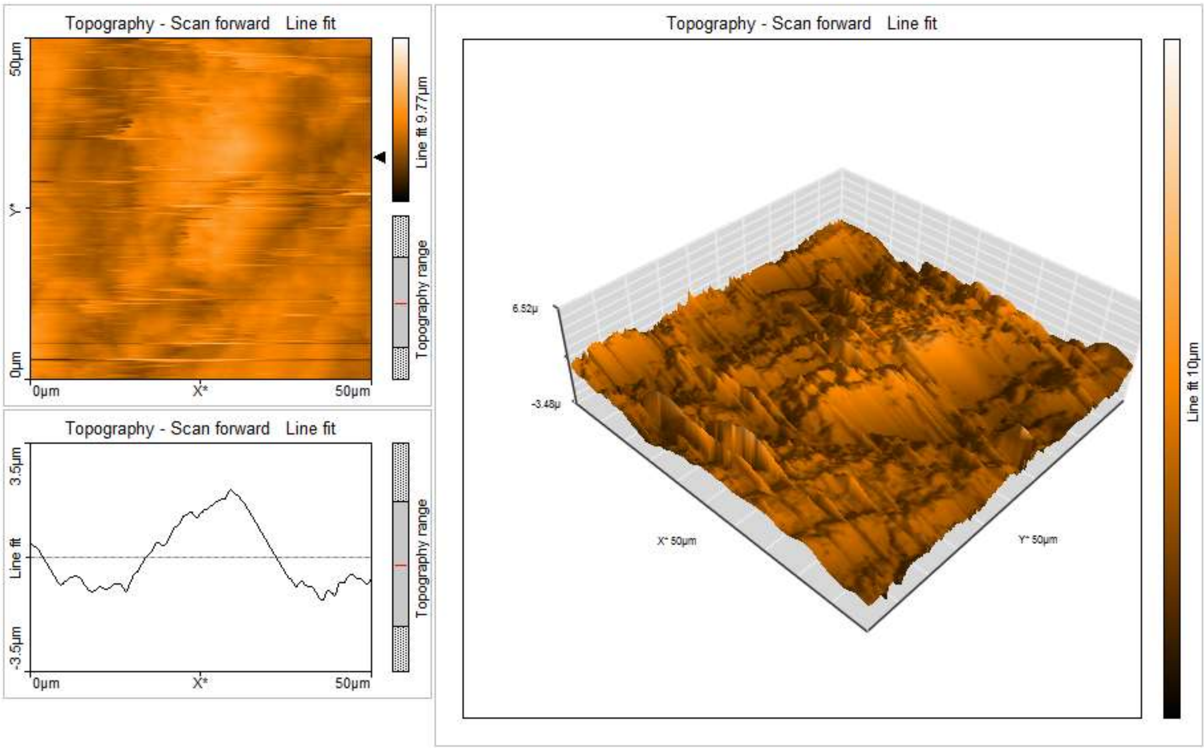


Figure - 6c: AFM cross sectional image for the zinc metal surface after immersion in 1N HCl with 10 ml of DSPL.

Conclusion

- The outcome of the study may find application in pickling industry
- In this present study, the anti-corrosion properties of an aqueous extract of *Datura stramonium* plant have been tested against the zinc metal in 1N HCl (corrosive medium).
- Increase in inhibition efficiency and decrease in corrosion rate of carbon steel in 1N HCl medium was observed with the increase in concentration of extract of DSP leaves. The maximum inhibition efficiency of 93.75% was achieved for 10 ml of *Datura stramonium* plant leaves.
- The potentiodynamic polarization studies conclude that *Datura stramonium* plant leaves performed as a anodic type of inhibitor.
- According to electrochemical impedance spectroscopy, there was an increase in polarization resistance (R_t) and decrease in double layer capacitance (C_{dl}). This behavior was found to be due to the formation of dense protective layer on the metal/electrolyte surface.
- The formation of protective film on the surface of zinc metal has been characterized by FTIR.
- Furthermore, microscopic studies such as SEM has indicated the presence of smooth surface in case of inhibited zinc metal when compared to the uninhibited samples.
- The roughness of zinc metal surface in the absence and presence of DSPLE inhibitor has been examined by Atomic Force Microscopy [AFM].

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