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Corrosion inhibitive action of an aqueous extract of *Datura candida* plant leaves on mild steel immersed in acid medium

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ABSTRACT

Corrosion inhibitive action of mild steel in 1M sulphuric acid by an aqueous extract of Datura candida plant leaves has been studied by mass loss method. The corrosion inhibition efficiency (IE) and corrosion rates are calculated from mass 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 mild steel surface. A maximum inhibition efficiency of 87.50% is achieved by this inhibitor at maximum concentration of 10 %. Potentiodynamic polarisation technique shows that the inhibitor system functions as an anodic type of inhibitor, controlling anodic reaction predominantly. Electrochemical studies indicates that a protective layer is formed on the mild steel surface. The surface morphology of the protective film has been studied by SEM. As a results of this present study will be helpful in acid pickling industry,

Keywords: *Acidic solutions, Datura candida plant leaves, Mass loss method, Mild steel corrosion and Scanning Electron Microscopy.*

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INTRODUCTION

Corrosion is the deterioration of a metal as a result of chemical reactions between it and the surrounding environment. Both the type of metal and the environmental conditions, particularly gasses that are in contact with the metal, determine the form and rate of deterioration. The use of sulphuric 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 [1-2]. 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. 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. Organic compounds containing electron donating groups or polar functional groups, heteroatoms, aromatic rings with π-electrons are widely used as effective corrosion inhibitors in industrial applications for various metals including mild steel. These inhibitors get adsorbed on the metal surface either chemically or physically forming a blanket on the metal surface, and thus isolate metal from the corrosive ions present in the medium. The use of inhibitors have been found to prevent the protection of mild steel against corrosion, especially in acidic media [3-4]. Inhibitors are used in the industrial process to control metal dissolution especially in acid, neutral and base environment. Many 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. [5-7]. The adsorption of inhibitor molecules on surface of carbon steel block the active sites of mild 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 [8-9]. The objective of the present study is to examine the aqueous extract of *Datura candida* plant leaves as inhibitor to control the corrosion of mild steel immersed in 1M H_2SO_4. The corrosion rate and inhibition efficiency has been evaluated by mass loss method. The mechanistic aspects of corrosion inhibition is determined by electrochemical studies such as polarization studies and alternating current impedance spectra. The protective layer was formed on 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).

MATERIAL 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 H_2SO_4) was prepared by dilution of an analytical grade sulphuric acid with double distilled water.

Preparation of inhibitor solutions

An aqueous extract of leaves of *Datura candida* 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.

Mass loss method

Mild steel specimens were immersed in 1M sulphuric 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 inhibitive action 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 [10].

AC impedance measurements

An electrochemical workstation impedance analyzer CHI- electrochemical work station with 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 Rt (charge transfer resistance) values were determined. The following relationship was used to measure C_{dl} values [11].

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

Surface Characterization 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 analysis techniques [SEM].

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 [12]. The SEM images were recorded by the SEM instrument, JEOL MODEL JSM 6390.

RESULT

Mass loss method

The present study is use to an aqueous extract of *Datura candida* plant leaves as inhibitor to control the corrosion of mild steel immersed in 1M H_2SO_4. The effectiveness of inhibitor in terms of corrosion rate and inhibition efficiency has been calculated by mass loss method. The corrosion rates (CR) of mild steel immersed in a 1M H_2SO_4 and also inhibition efficiencies (IE) in the absence and presence of the extract of the *Datura candida* inhibitor obtained by mass loss method are given in Table.1. It is observed that 10% of the extract of *Trifolium pratense* offers 87.50 % of inhibition efficiency [13].

Analysis of results 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 [14].

The potendiodynamic polarization curves of mild steel immersed in 1M sulphuric 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 results of alternating current impedance spectra

The formation of a protective layer on the mild steel surface has been confirmed using alternating current impedance spectra (electrochemical impedance spectra) [15-17]. As a protective layer is formed on the mild steel surface, the charge transfer resistance (Rt) increases, the double layer capacitance value (Cdl) decreases, and the impedance log(z/ohm-1) value increases. The AC impedance spectra of mild steel immersed in 1M H_2SO_4 in the absence and presence of inhibitor are shown in Figure 2a, Figure 2b, Figure 3b and Figure 3b and the values are given in Table 3.

SEM Analysis of mild steel surface

To understand the surface condition of the studied mild steel specimens, its surface morphology has been examined by scanning electron microscope. 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 [18-19].

DISCUSSION

Analysis of mass loss method

It is observed from Table 1 that as the concentration of the extract of *Datura candida* increases, the inhibition efficiency increases and corrosion rate decreases. This is due to an increase of surface coverage at higher concentration of the *Datura candida* which retards dissolution of mild steel. A protective layer is formed on the mild steel surface. It consists of Fe^{2+} - active principles (present in the extract) complex [20-21]. The possibility of interaction between the hetero atoms [oxygen] 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 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.

Table 1: Inhibition efficiency of aqueous extract of *Datura candida* plant leaves in controlling corrosion of mild steel in 1M H_2SO_4.

Concentration of plant extract (ml)	Corrosion rate (mdd)	Inhibition Efficiency (%)
Blank	376.20	-
2	195.50	49.90
4	134.70	62.10
6	102.80	72.80
8	87.90	79.60
10	27.10	87.50

Analysis of results of potentiodynamic polarization study

When mild steel was immersed in 1M sulphuric acid the corrosion potential was -608 mV vs SCE. When 10% of aqueous extract of *trifolium pratense* plant leaves was added to the above system, the corrosion potential was shifted to the anodic side -468 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 [22-23].

Further, the LPR value increases from 405 ohm.cm^2 to 888 ohm.cm^2, the corrosion current decreases from 1.022×10^{-4} to 4.804×10^{-5} (A/cm^2). Thus polarization study confirms the formation of a protective film on the mild steel surface.

Table 7. Corrosion parameter of mild steel immersed in 1M sulphuric acid in the absence and presence of inhibitor system obtained by potentiodynamic polarization method.

Concentration of the aqueous extract of DCPL (mL)	E_{corr} vs SCE (mV)	I_{corr} (A/cm^2)	b_a (mV/dec)	b_c (mV/dec)	LPR (ohm. cm^2)
0	- 530	1.224×10^{-6}	471	510	36302
10	- 481	1.237×10^{-9}	478	507	35698904

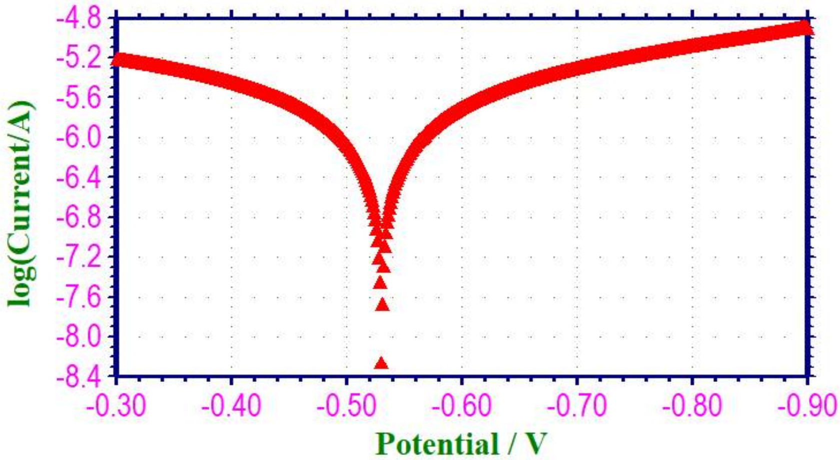

Figure 1a. Potentiodynamic polarization curves of mild steel in 1M H_2SO_4 (blank)

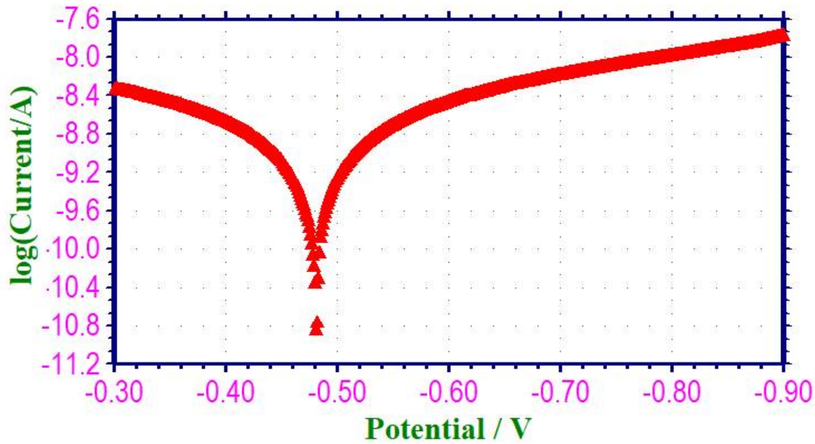

Figure 1b. Potentiodynamic polarization curves of mild steel in 1M H_2SO_4 (blank) + 10 ml of plant extract

Analysis of results of alternating current impedance spectra

It is observed that when the inhibitor (10 ml of plant extract) is added to the above system, the charge transfer resistance (Rt) increases from 2184 Ω cm^2 to 12483330 Ω cm^2 and the Cdl value decreases from 3597.048×10^{-6} Fcm^{-2} to $20560044.51 \times 10^{-7}$ Fcm^{-2}.The impedance value [log (z/ohm^{-1})] increases from 0.784 to 2301. These findings point to the formation of a protective layer on the mild steel surface [24-25].

Table 4. Corrosion parameters of mild steel immersed in 1M H_2SO_4 solution in the absence and presence of inhibitor system obtained from AC impedance spectra

Systems	Impedance		
	R_t Ωcm^2	C_{dl} F/cm^{-2}	Log (Z ohm^{-1})
1M H_2SO_4 (blank)	2184	3597.048×10^{-6}	0.784
1M H_2SO_4 + 10 ml of plant extract	12483330	$20560044.51 \times 10^{-7}$	2301

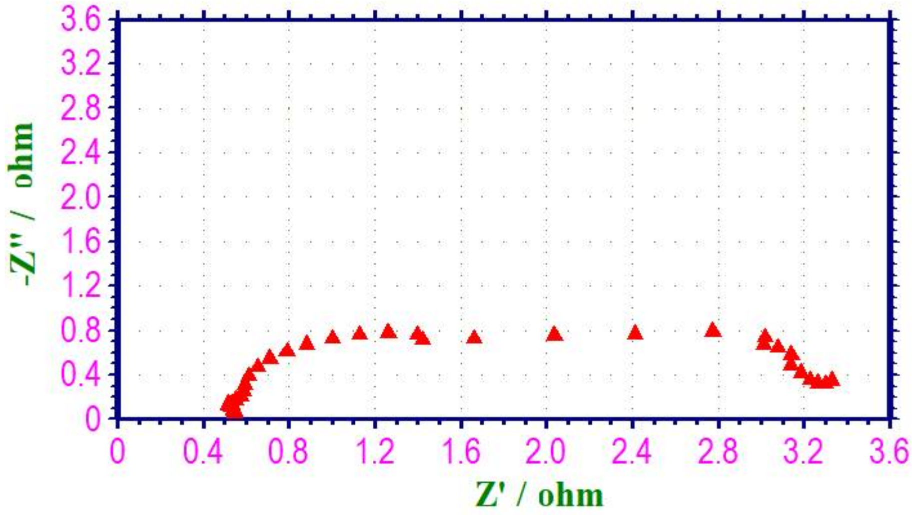

Figure 2a. Electrochemical impedance curve (Nyquist plot) of mild steel in 1M H_2SO_4 (blank)

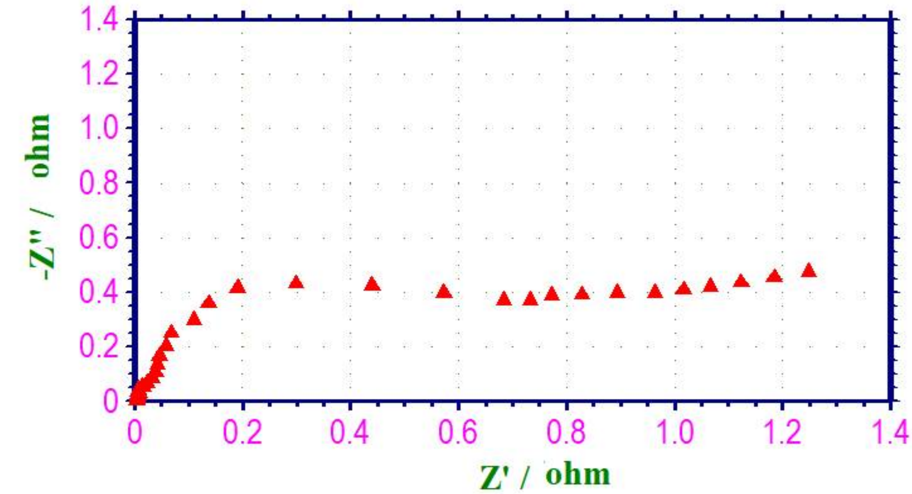

Figure 2b. Electrochemical impedance curve (Nyquist plot) of mild steel in 1M H_2SO_4 (blank) + 10 ml of plant extract.

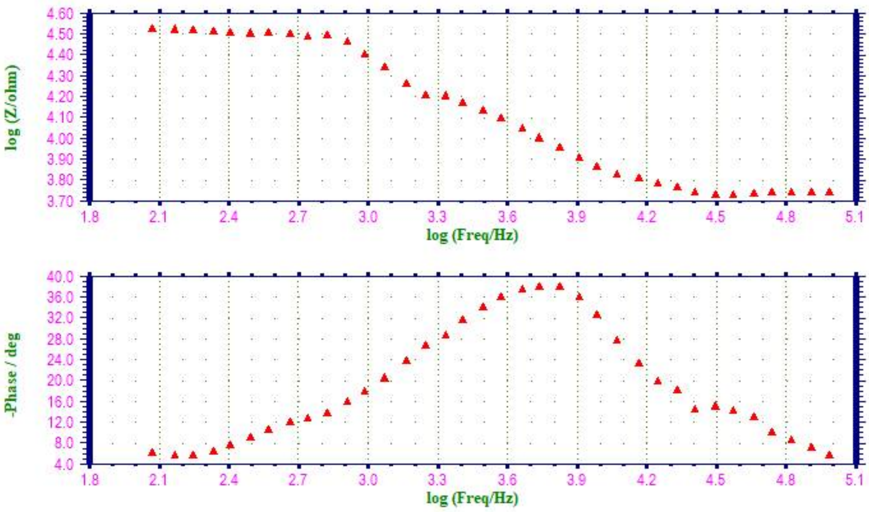

Figure 3a. Electrochemical impedance curve (Bode plot) of mild steel in 1M H_2SO_4 (blank)

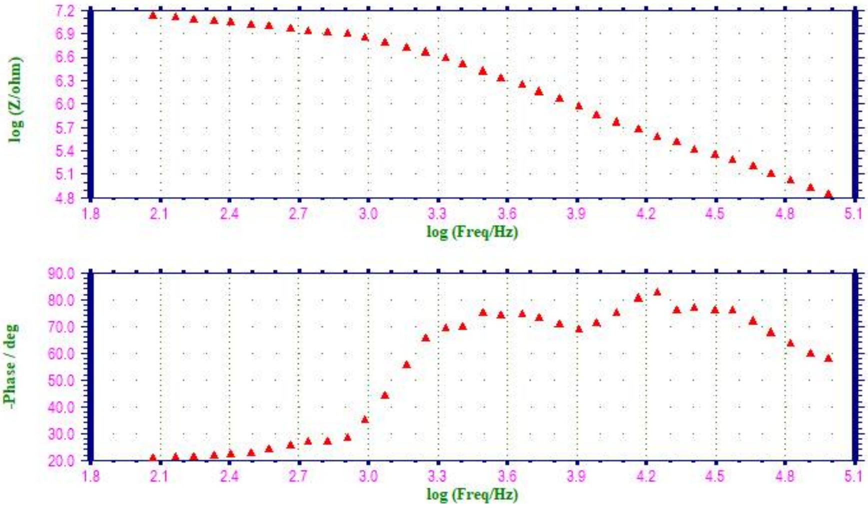

Figure 3b. Electrochemical impedance curve (Bode plot) of mild steel in 1M H_2SO_4 (blank) + 10 ml of plant extract.

Analysis of mild steel surface by SEM

The SEM images of mild steel specimens immersed in 1M H_2SO_4 for two hours in the absence and presence 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 H_2SO_4 for 2 hours. Figure 4c shows the SEM image of polished mild steel immersed in 1M H_2SO_4 and inhibitor system for a period of 2 hours. 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 on the mild steel surface.

The SEM micrograph of mild steel surface immersed in 1M H_2SO_4 (Figure 4b) shows the roughness of the mild steel surface which indicates the highly corroded surface of mild steel in 1M H_2SO_4. However, Figure 4c indicates that in the presence of inhibitor (10 ml of plant extract) 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 DCPL, the surface is covered by a thin layer of inhibitors which effectively controls the dissolution of mild steel [26-28].

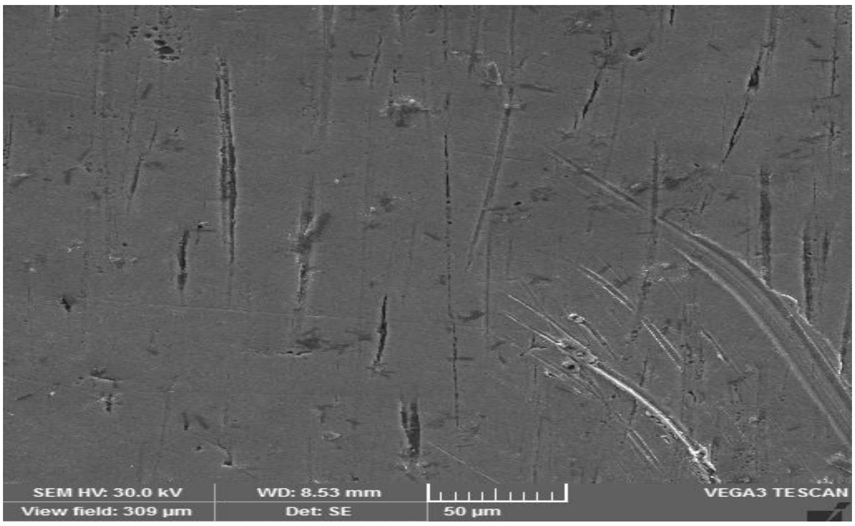

Figure 4a. SEM image of polished mild steel coupon before immersion in 1M H_2SO_4 (control)

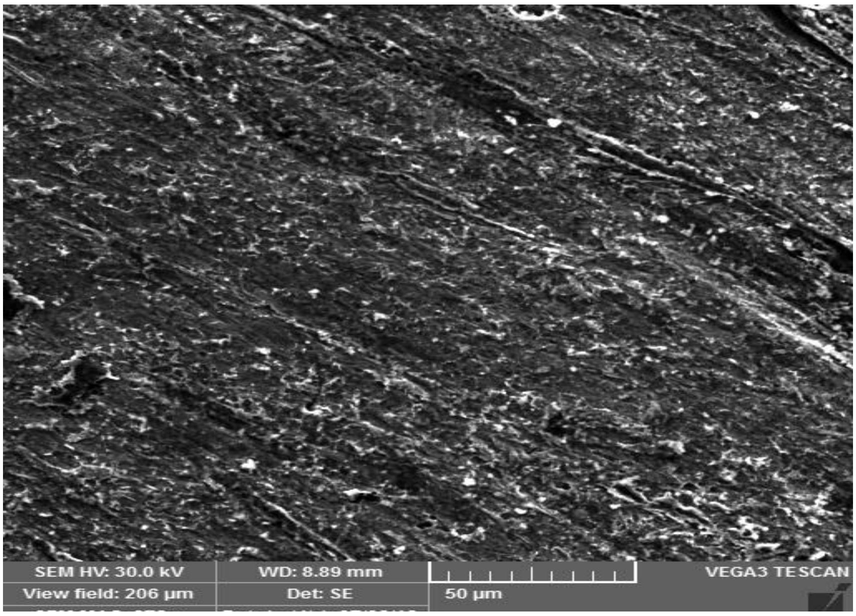

Figure 4b. SEM image of mild steel coupon after immersion in 1M H H_2SO_4 (blank)

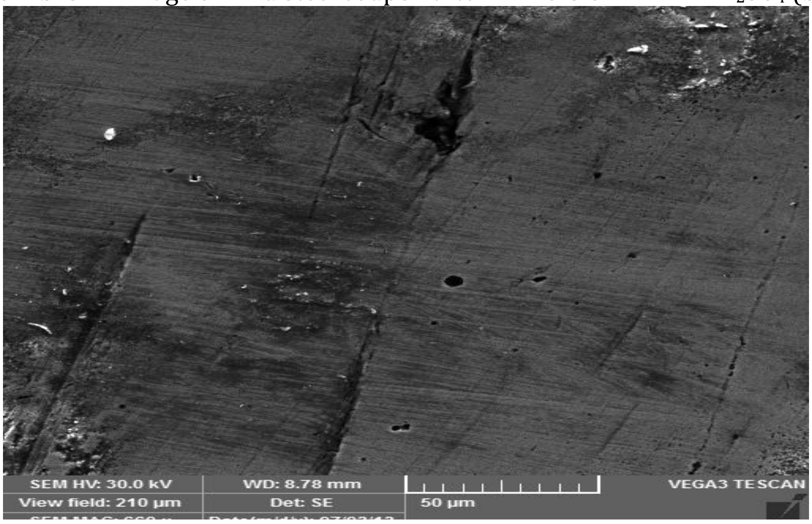

Figure 4c. SEM image of polished mild steel coupon after immersion in 1M H_2SO_4 in the presence of 10 ml of an aqueous extract of DCPL.

CONCLUSION

The inhibitive influence of leaves of *Datura candida* on the corrosion of mild steel in 1M H_2SO_4 medium was studied by mass loss method, polarization and impedance measurements. The corrosion is decreased with increase in the addition of *Datura candida* leaves extract probably due to the progressive adsorption of theinhibitor on the mild steel surface. The maximum inhibition efficiency was found to be 87.50%. The corrosion of mild steel in 1M H_2SO_4solution is inhibited by the addition of leaves extract of *Datura candida*. The percentage of inhibition efficiency increases with increase of the inhibitor concentration. The corrosion of mild steel inhibited by leaves extract of *Datura candida* attributed to the adsorption of phytochemical components present in the inhibitor onto the mild steel surface. The results obtained suggest that the *Datura candida* leaves extract is a good corrosion inhibitor for mild steel in 1.0 M H_2SO_4medium and it can be preferentially used to replace toxic and non-biodegradable inhibitor. Electrochemical measurements indicate that an increase the charge transfer resistance (R_t), decrease the double layer capacitance (C_{dl}) and corrosion current ($_{Icorr}$) values owing to the increased thickness of adsorbed layer. SEM images show the smoothness of mild steel surface like polished mild steel.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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