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Evaluation of Corrosion Resistance of an Aqueous Extract of *Trifolium pratense* plant leaves on mild steel immersed in acidic media

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ABSTRACT
Evaluation of corrosion resistance of mild steel in 1M hydrochloric acid by an aqueous extract of leaves of Trifolium pratense plant has been studied by weight loss method. The corrosion inhibition efficiency (IE) and corrosion rates are calculated from 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 85.20% is achieved by this inhibitor system. Temperature study is used to determine the mechanism and percentage inhibition of these additives. In this case, the corrosion efficiency and corrosion rate is determined for TPPLE inhibitor system from 2 - 10% v/v with temperatures ranging from 303-333 K. The effect of temperature increased with increasing inhibitor concentration on the dissolution of mild steel and the partial desorption of with inhibitor molecule from the surface of the mild steel. Several isotherms have been formulated for the adsorption of the corrosion inhibitor molecules on the surface of mild steel. From the isotherm, the linear relationship between θ and concentration of inhibitor can be found. In this study, the changes in the θ and thereby the change in the efficiency of inhibitor is determined by using different isotherms model. Potentiodynamic polarisation technique reveals that the inhibitor system functions as an anodic type of inhibitor, controlling anodic reaction preferably. 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. 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, *Trifolium pratense* plant leaves, Mild steel corrosion, Scanning Electron Microscopy and Weight loss method.

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INTRODUCTION

Mild steel finds applications in automobile body components, structural shapes, sheets, etc. 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]. 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. 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. Some 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 (MS). 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 has been found to prevent the protection of carbon steel against corrosion, especially in acidic media [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]. The objective of the present study is to evaluate the aqueous extract of *Trifolium pratense* 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 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).

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 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 *Trifolium pratense* 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 [11-12].

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

RESULT

Weight loss method

The present study an aqueous extract of *Trifolium pratense* 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 *Trifolium pratense* inhibitor obtained by weight loss method are given in Table.1. It is observed that 10% of the extract of *Trifolium pratense* offers 85.20 % of inhibition efficiency.

Effect of temperatures

Study the effect of temperature on the corrosion rate by chemical method (weight loss method) will be enable us to calculate the thermodynamic parameters related to the corrosion process. It is also useful to determine the kind of adsorption isotherms and the type of adsorption (physical or chemical).

Adsorption isotherm

An adsorption isotherm is a mathematical expression of graphical curve which represents the relation between the bulk concentrations of an adsorbing species to its surface concentration at constant temperature. An adsorption isotherm gives the relationship between the coverage of an interface with an adsorbed species (the amount adsorbed) and the concentration of the species in solution [16]. The adsorption isotherm provide the basic information about the nature of interaction between the mild steel surface and an inhibitor molecular constituents [17].

Langmuir adsorption isotherm

Most of the organic inhibitors and plant extracts obey the Langmuir adsorption isotherm. The degree of surface coverage (Θ) for different concentrations of inhibitor (2, 4, 6, 8 and 10 % (v/v)) at different temperatures (303, 313, 323 and 333 K) has been found from weight loss method. According to Langmuir isotherm, the following equation relates the surface coverage (Θ) and the inhibitor concentration C.

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C$$

Temkin adsorption isotherm

The values of surface coverage Θ, at various concentrations of TPPLE inhibitor in 1M HCl solution obtained from weight loss measurements, were fitted to Temkin adsorption isotherm shown below. The Temkin adsorption isotherm is based on the assumption of uniform distribution of the TPPLE inhibitor (monolayer) on the mild steel surface [31]. The plot of ΘVs.log C for the mild steel in 1M HCl in the absence and in the presence of inhibitor system TPPLE is given in the **(Figure. 2)** for different temperatures.

Free energy adsorption (ΔG^0_{ads})

The free energy of adsorption, ΔG^o_{ads} at different temperature are determined by Langmuir adsorption isotherm (best fit) isotherm model using the equation,

$$\Delta G^\circ_{ads} = -2.303RT \log (K_{ads} \times 55.55)$$

Energy of activation (E_a)

The apparent activation energy for the corrosion samples in 1M HCl solution in the absence and presence of different concentration of *trifolium pratense* plant leaves were calculated from Arrhenius type equation [34]. The Arrhenius type is given below

$$\log CR = K_{exp} (-E_a / RT)$$

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 [37].

The potendiodynamic polarization curves of mild steel immersed in 1M hydrochloric acid in the absence and presence of inhibitor are shown in Figure 5 (a, b). The corrosion parameters are given in Table 7.

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 [39-40].

DISCUSSION

Analysis of weight loss method

It is observed from Table 1 that as the concentration of the extract of *Trifolium pratense* increases, the IE increases. This is due to an increase of surface coverage at higher concentration of the *Trifolium pratense* 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 [14-15]. 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 *Trifolium pratense* 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	146.20	45.00
4	114.10	57.10
6	67.80	68.50
8	42.80	80.90
10	21.40	85.20

Analysis of effect of temperature

The data of inhibition efficiency and the corrosion rate of TP plant leaves extract are given in table - 2. The inhibition efficiency is decreases from 85.20 to 73.70% and the corrosion rate is increases from 21.40 to 142.70 as the rise in temperature from 303 – 333 K in 1M HCl at the higher concentration (10%) for TPP leaves extract. The effect of temperature increased with increasing inhibitor concentration on the dissolution of mild steel and the partial desorption of with inhibitor molecule from the surface of the mild steel.

Table – 2. Inhibition efficiency of aqueous extract of TPPL on the corrosion of mild steel in 1M HCl at different temperatures

Temperature (K)	Concentration of Inhibitor (% v/v)	Corrosion rate (mmd)	Inhibition efficiency (%)
303	blank	265.70	-
	2	146.20	45.00
	4	114.10	57.10
	6	67.80	68.50
	8	42.80	80.90
	10	21.40	85.20
313	blank	328.50	
	2	206.80	37.60
	4	181.90	51.10
	6	139.10	62.00
	8	89.20	76.10
	10	46.40	88.10
323	blank	355.20	-
	2	246.10	30.70
	4	178.30	49.80
	6	149.80	57.80
	8	96.30	72.90
	10	85.90	85.90
333	blank	542.10	-
	2	392.30	27.60
	4	321.00	40.80
	6	267.50	50.70
	8	185.50	65.80
	10	142.70	73.70

Langmuir adsorption isotherm

The plots of Langmuir adsorption isotherm, C/Θ Vs. C at different temperatures is shown in **(Figure. 1)** for TPPLE studied inhibitor at different temperatures.

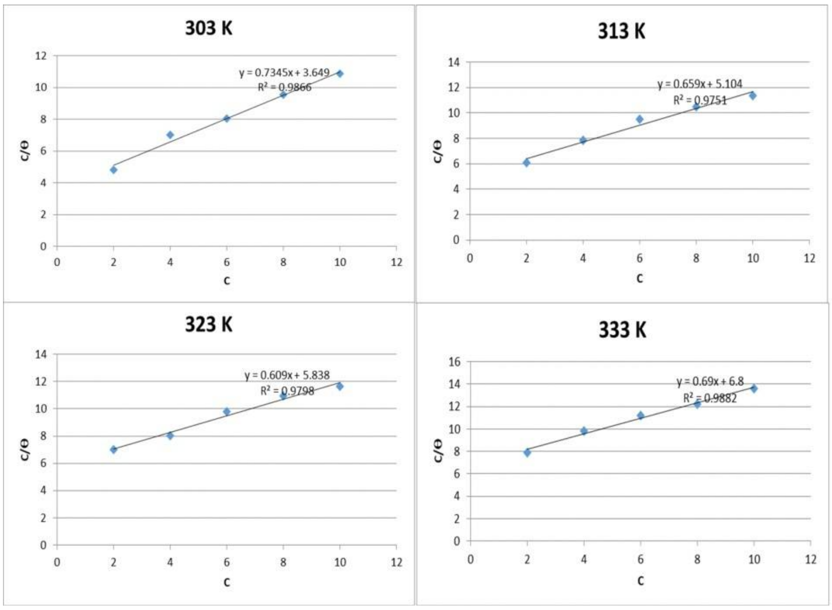

Figure - 1 Langmuir adsorption isotherm curves for the adsorption of TPPLE on mild steel in 1M HCl at different temperatures.

Adsorption parameters on mild steel corrosion inhibition by TPPLE system obtained from Langmuir adsorption isotherm, including ΔG^0_{ads}, R^2, slope, intercept and the parameters are given in **Table –3.**

Table – 3: Adsorption parameters obtained from Langmuir adsorption isotherm for the corrosion inhibitive effect of aqueous extract of TPPL on the corrosion of mild steel in 1M HCl

Inhibitor system	Temperature (K)	R^2	Slope	Intercept	K_{ads}	$-\Delta G^0_{ads}$ KJ mol^{-1}
Aqueous extract of TPPL	303	0.9866	0.7345	3.649	0.274	6.90
	313	0.9751	0.659	5.104	0.4753	8.517
	323	0.9829	0.609	5.838	0.171	6.044
	333	0.9882	0.690	6.80	0.147	5.812

The spontaneity of adsorption process and the stability of adsorbed layer on the mild steel surface were confirmed by the negative values of ΔG^0_{ads}. The ΔG^0_{ads} is calculated from the equation

$$- \Delta G^o_{ads} = - 2.303\ RT \log (K_{ads} \times 55.55)$$

The slopes of the plots are expected to be unity on the assumption that there is no interaction among the adsorbed inhibitor molecules and the surface of the mild steel for the ideal Langmuir adsorption isotherm [18-20]. But the slope values deviate from unity suggesting the presence of interaction among the adsorbed inhibitor molecules on the mild steel surface. The inhibitor molecules adsorbed on the anodic and cathodic sites of the mild steel surface interact by mutual repulsion (or) attraction [21-28]. The comparison of R^2 values obtained from Langmuir adsorption isotherm, at various temperatures shows that the isotherm fits better at 303 K than the other higher temperatures and it has been reported by many researchers [29]. The R^2 values are greater than 0.9 showing that the Langmuir plots are linear. The K_{ads} values are high at 303 K for the inhibitor system TPPLE and decrease with the increasing the temperatures, it indicating that the inhibitor is more strongly adsorbed on the carbon steel surface at low temperature than at higher temperatures [30].

Temkin adsorption isotherm

The plot of ΘVs.log C for the mild steel in 1M HCl in the absence and in the presence of inhibitor system TPPLE is given in the **(Figure. 2)** for different temperatures.

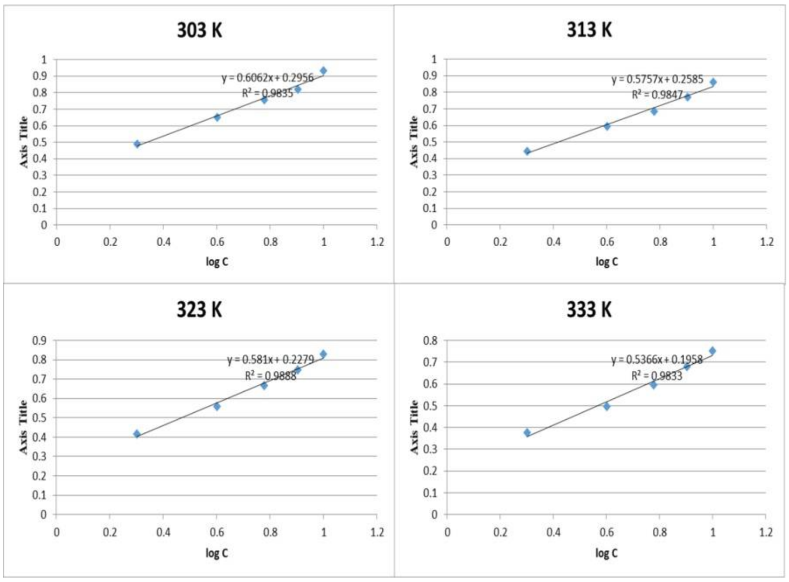

Figure - 2 Temkin adsorption isotherm curves for the adsorption of TPPLE on mild steel in 1M HCl at different temperatures.

From Temkin isotherm plots, the values of intercept, slope, a, K_{ads} and ΔG^0_{ads} are calculated and shown in Table – 4.

Table – 4: Adsorption parameters obtained from Temkin adsorption isotherm for the corrosion inhibitive effect of aqueous extract of TPPL on the corrosion of mild steel in 1M HCl

Inhibitor system	Temperature (K)	R^2	Slope	Intercept	-a	K_{ads}	$-\Delta G^0_{ads}$ KJ mol^{-1}
Aqueous extract of TPPL	303	0.9835	0.6062	0.2956	1.899	3.072	12.947
	313	0.9847	0.5759	0.2585	1.999	2.810	13.142
	323	0.9847	0.5757	0.2585	2.000	2.812	13.564
	333	0.9888	0.581	0.2279	1.982	2.468	13.623

The negative ΔG^o_{ads} values indicate that the adsorption of inhibitor molecules from the aqueous extract of BRPL on the mild steel surface is spontaneous. The ΔG^o_{ads} values up to -20 kJ mol^{-1} are consistent with electrostatic interaction between the charged molecules and the charged metal (physisorption) and while those more negative than -40 kJ involve charge sharing or transfer of electrons from the inhibitor molecules to the metal surface to form a coordinate type of bond (chemisorption) [32]. The high values of K_{ads} and low negative ΔG^0_{ads} values at temperature ranging from 303-303K (Table – 5) shows that the physisorption occurs. However, chemisorption may not be excluded due to the nature of complex formation in the corrosion inhibiting process.

Free energy adsorption (ΔG^0_{ads})

The evaluated mean value of ΔG^o_{ads} at different temperatures is given in Table 5.

The negative value of ΔG^o_{ads} calculated from Langmuir adsorption isotherm indicate the occurrence of spontaneous adsorption of inhibitor molecules from the plant extracts on the mild steel surface. The ΔG^0_{ads} values are up to -20 kJ mol^{-1} and the values are consistent with electrostatic interaction between the charged molecules and the charged metal are known as physisorption [33].

Table – 5: Average free energy of adsorption of mild steel at different temperatures in 1M HCl in the presence of aqueous extract of TPPL.

Inhibitor system	$-\Delta G°_{ads}$ (kJ/mol^{-1})			
	303K	313K	323K	333K
Aqueous extract of TPPL	12.947	13.142	13.56	13.623

Energy of activation (E_a)

The values of E_a, is calculated for mild steel immersed in the absence and in the presence of TPPLE system, from the slope value of **logCR Vs.1/T**plots **(Figure. 3 and 4)** and the $\mathbf{E_a}$ values are shown in **Table –6.**

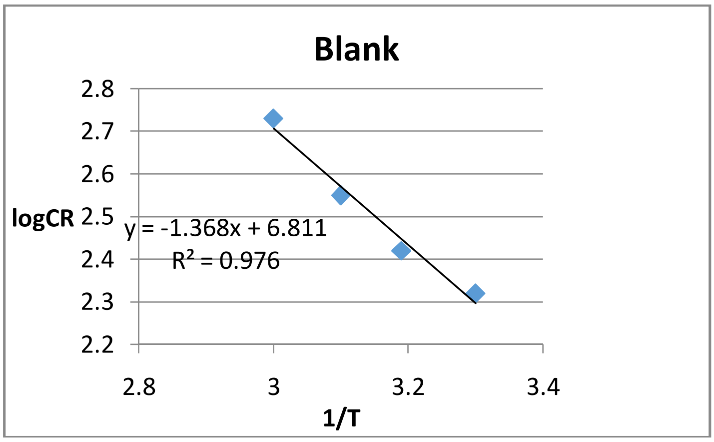

Figure –3: Arrhenius plot for mild steel in 1M HCl without the inhibitor system (blank)

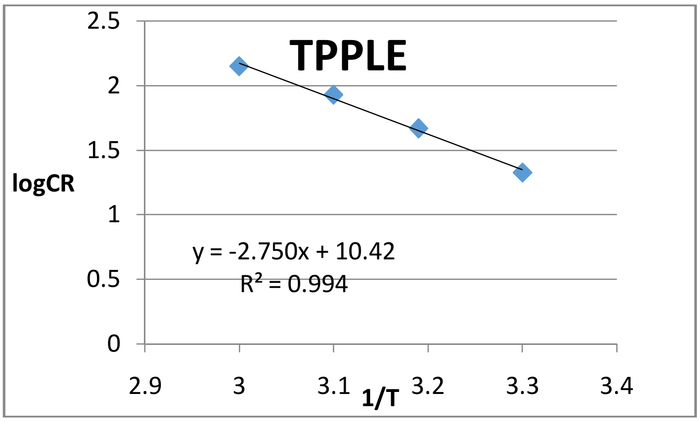

Figure –4: Arrhenius plot for mild steel in 1M HCl with 10% aqueous extract of TPPL inhibitor.

The calculated E_a values are high for the system with maximum inhibitor concentration than to the system without inhibitor. From this it is suggested that at low temperature TPPLE inhibitor found to be more effective. Hence the inhibition efficiencies become less and the corrosion rate will more at high temperatures [35]. Because the desorption takes place, greater surface area of mild steel comes in contact to the environment at higher temperature. But this problem is reduced in low temperature because of the degree of surface coverage of carbon steel become close saturation [36].

Table – 6: Average activation energy for mild steel corrosion of in 1M HCl in the absence and presence of aqueous extract of TPPL (10% v/v).

System	E_a (KJ/mol)
Blank	26.20
Aqueous extract of TPPL	52.67

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 *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 [38]. 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 hydrochloric acid in the absence and presence of inhibitor system obtained by potentiodynamic polarization method.

Concentration of the aqueous extract of TPPL (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	- 468	4.804×10^{-5}	210	183	888

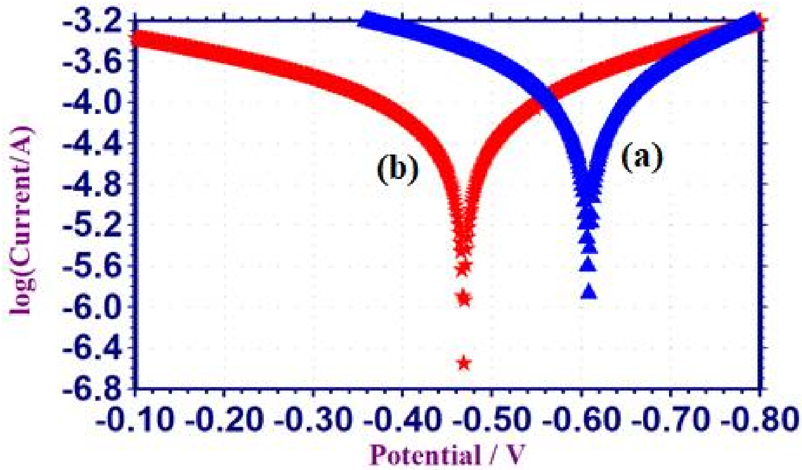

Figure 5. Potentiodynamic polarization curves of mild steel in 1M HCl (a) in 1M HCl (blank) (b) in 1M HCl + inhibitor extract

SEM Analysis of mild steel surface

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 6 (a, b and c). Figure 6a shows the SEM image of polished mild steel surface. Figure 6b shows the SEM image of polished mild steel immersed in corrosive medium, namely 1M HCl for 2 hours. Figure 6c shows the SEM image of polished mild steel immersed in 1M HCl and inhibitor system for a period of 2 hours. The SEM micrographs of polished mild steel surface (control) in Figure 6a 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. The SEM micrograph of mild steel surface immersed in 1M HCl (Figure 6b) shows the roughness of the mild steel surface which indicates the highly corroded surface of mild steel in 1M HCl. However, Figure 6c indicates that in the presence of inhibitor (10% of TPPL) 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 BRPL, the surface is covered by a thin layer of inhibitors which effectively controls the dissolution of mild steel [41].

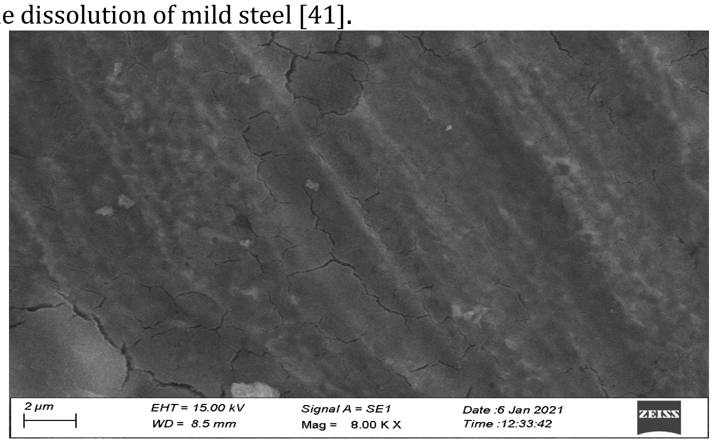

Figure 6a. SEM image of polished mild steel specimen before immersion in 1M HCl (control)

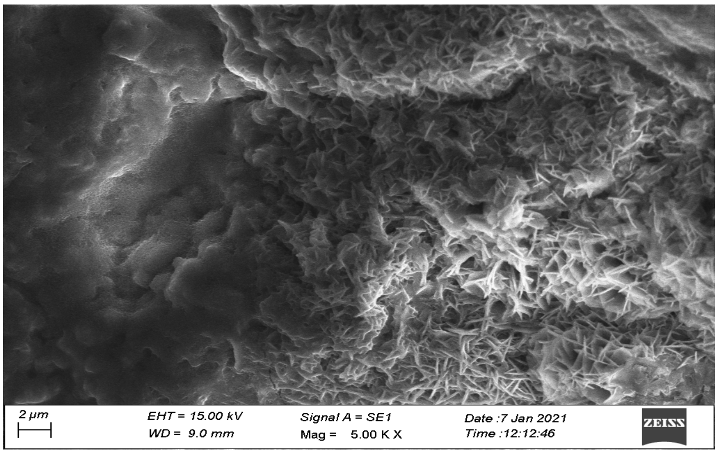

Figure 6b. SEM image of mild steel specimen after immersion in 1M HCl (blank)

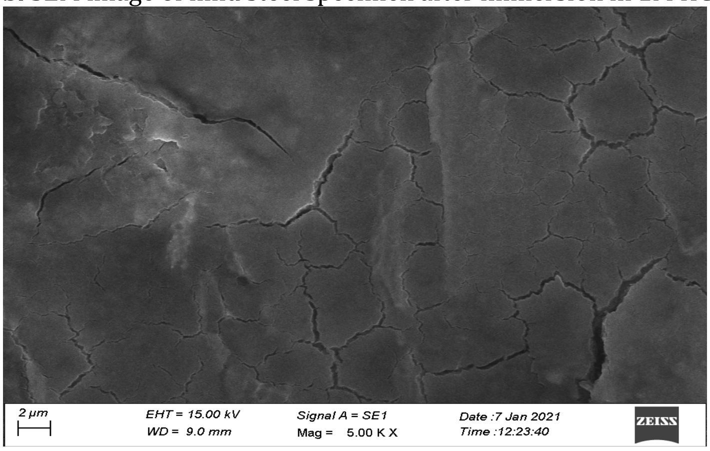

Figure 6c. SEM image of polished mild steel specimen after immersion in 1M HCl in the presence of 10% of an aqueous extract of TPPL.

CONCLUSION

In this study the aqueous leaves extract of *trifolium pratense* plant 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 *trifolium pratense* 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 *trifolium pratense* systems function as anodic inhibitor controlling the anodic reaction predominantly.
- The weight loss technique shows the inhibition efficiency is 85.20%.
- 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 micrographs show the smoothness of mild steel surface

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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