

THE STUDY OF HENNA LEAVES EXTRACT AS GREEN CORROSION INHIBITOR FOR MILD STEEL IN ACETIC ACID

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ABSTRACT

The inhibitive action of henna (*Lawsonia Inermis*) leaves on mild steel in acetic acid solution have been investigated by weight-loss, A C impedance and potentiodynamic polarization measurements. The study indicates that as acid concentration increases corrosion rate increases. The corrosion inhibition efficiency increases with increase in concentration of extract. The result obtained revealed that henna leaves extract act as an efficient inhibitor. The adsorption of the henna leaves extract obeyed Langmuir adsorption isotherm. The calculated thermodynamic parameters indicated that the adsorption was a spontaneous, exothermic process accompanied by an increase in entropy. Cathodic and anodic polarization curves show that henna leaves extract is a mixed-type inhibitor.

Key words: corrosion; mild steel; acetic acid; henna leaves extract; green inhibitor.

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1. INTRODUCTION

Corrosion of metal/alloy, which can be define as the deterioration of materials due to their reaction with the environment. It is a common problem in industry and it has received a considerable amount of attention. Mild steel is widely used as constitutional material in many industries due to its good mechanical property and low cost. However, the main problem using mild steel is its dissolution in acidic medium. It is found that organic acids are present in produced water and was reported as a several concern in corrosion of mild steel pipeline in oil and gas industries [1-4]. Generally lower molecular weight organic acids can lead to corrosion of mild steel pipeline [5]. The use of inhibitor is one of the most important methods for protection of mild steel against corrosion in acid media. The extract of plant leaves also known as green inhibitors such as *Newbouldia leavis* [6], *Carica Papaya* and *Camellia Sinensis*[7], *Euphorbia hirta* [8], *Tithonia diversifolia* [9], *Cola Acuminata* and *Camellia Sinensis* [10], *Vernonia Amygdalina* [11,12] among other have been studied for corrosion inhibition of metal in various media. All these studies reveals that the plant extract contain organic compound like those contains N, S and O showed inhibition efficiency. The main aim of present work is to find out corrosion inhibition efficiency (I.E.) of aqueous extract of henna leaves as green inhibitor for mild steel in acetic acid solution.

2. RESULTS AND DISCUSSION

2.1. Weight loss measurement

Effect of different concentration (1, 0.5, 0.25 and 0.1 g/lit) of henna leaves extract was studied in 0.5, 1.0, 1.5 and 2.0 M acetic acid solution at room temperature of 303 ± 1 K for exposure period of 24 h.

The obtained results are presented in Table-1. The corrosion loss has been increased from 486.41 to 891.75 mg/dm^2 in 1.0 to 2.0 M acetic acid solution. It has been observed that the corrosion rate increases with increase in the acid concentration. It can be concluded that corrosion rate is directly proportional to the acid concentration.

Table 1. Effect of acid concentration on corrosion loss (CL) and inhibition efficiency (I.E.) of henna leaves extract on mild steel

Inhibitor Concen- tration (g/lit)	Acid concentration							
	0.5 M		1.0 M		1.5 M		2.0 M	
	CL mg/dm ²	I.E. %	CL mg/dm ²	I.E. %	CL mg/dm ²	I.E. %	CL mg/dm ²	I.E. %
Blank	486.41		629.47		767.76		891.75	
0.1	119.22	75.49	157.37	75.00	195.52	74.53	224.13	74.86
0.25	95.37	80.39	123.99	80.30	152.60	80.12	181.21	79.68
0.5	81.07	83.33	109.68	82.26	133.52	82.60	157.37	82.35
1.0	66.76	86.27	85.84	86.36	104.92	86.33	123.99	86.09

Effective area of specimen: 0.2097 dm² Immersion period: 24 h Temp.: 303 ± 1 K

The I.E. increases as the follow:

$$IE\% = \frac{W_u - W_i}{W_u} \times 100 \quad \text{----- (1)}$$

Where, w_u and w_i are the weight-loss of the metal in uninhibited and inhibited acid respectively. The I. E. increases as the inhibitor concentration increases.

Adsorption isotherm and thermodynamic measurements:

In order to study the adsorption of henna leaves extract, on mild steel surface, best agreement was obtained with Langmuir adsorption isotherm. Langmuir adsorption isotherm is an ideal isotherm for physical and chemical adsorption [13,14] as well, This isotherm can be represented as,

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad \text{----- (2)}$$

Where, K_{ads} is the equilibrium constant of the adsorption process and C_{inh} is the inhibitor concentration. The value of surface coverage θ ($\theta = W_u - W_i / W_u$) were calculated directly from the percentage I. E. of the compounds by weight-loss method.

The plot of extract concentration C versus C/θ was shown in Fig.1, straight line with almost unit slope was obtained indicating that the system follows Langmuir adsorption isotherm [13].

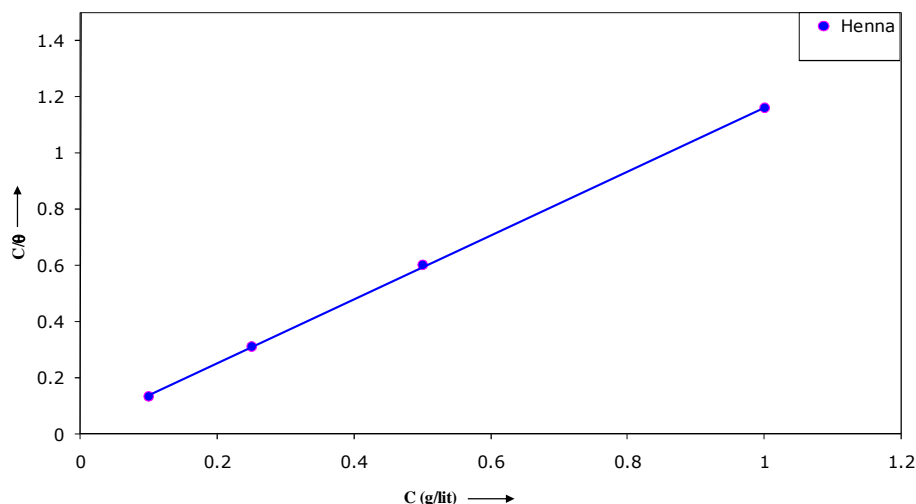


Fig.1. Langmuir adsorption isotherm for henna leaves extract in 0.5 M acetic acid

In order to get basic information about the interaction between the inhibitor molecule and mild steel surface the Langmuir adsorption isotherm is also established in the range of studied temperature. The correlation between the extract concentration and surface coverage is given Fig.2. The linear plot of C versus C/θ for each temperature has slope value close to one and the K_{ads} was obtained from the intercept. The standard free energy of activation $\Delta G^{\circ}_{\text{ads}}$ is related to K_{ads} according to following equation [15].

$$K_{\text{ads}} = \frac{1}{C_{\text{solvent}}} \exp\left(\frac{-\Delta G^{\circ}_{\text{ads}}}{RT}\right) \quad \text{----- (3)}$$

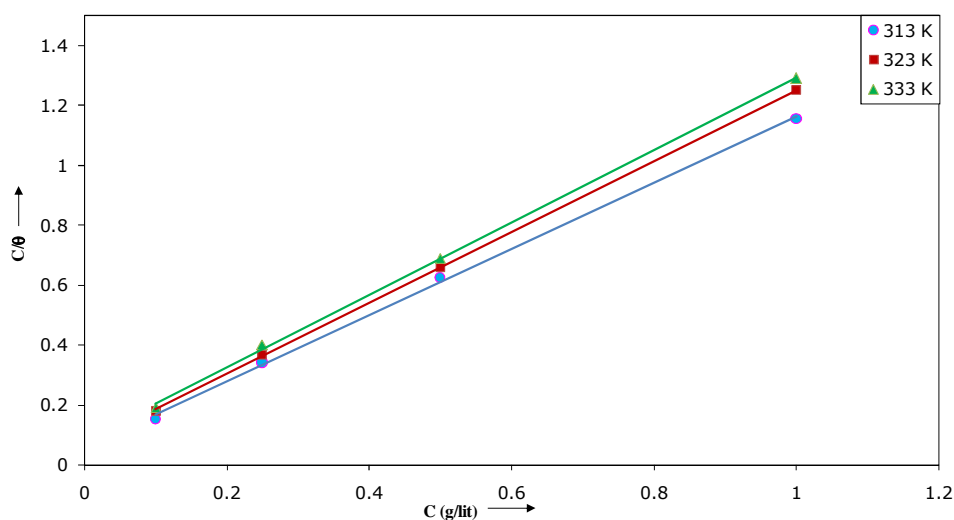


Fig.2. Langmuir adsorption isotherm plot for the adsorption of henna leaves extract in 1.0 M acetic acid solution at different temperature

The negative value of $\Delta G^\circ_{\text{ads}}$ suggest that adsorption of henna leaves extract on mild steel surface is spontaneous. As the value of $\Delta G^\circ_{\text{ads}}$ ranges from -17.99 to -18.12 kJ mol⁻¹ concluded that the extract is physically adsorbed on the mild steel surface and thus create an electrostatic interaction [16]. Generally the around -20 kJ mol⁻¹ of $\Delta G^\circ_{\text{ads}}$ value is associated with electrostatic interaction between charge molecule and and the charged metal surface. (physisorption), while the higher than -40 kJ mol⁻¹ refer to charge sharing or transfer from the inhibitor molecules to the metal surface to form a coordinate type of bond (chemisorption) [17].

The heat of adsorption $\Delta H^\circ_{\text{ads}}$ can be calculated from the Vant Hoff equation [18] as follows:

$$\ln K_{\text{ads}} = \frac{-H^\circ_{\text{ads}}}{RT} + \text{constant} \quad \text{----- (4)}$$

Where R is gas constant and T is absolute temperature.

The $\Delta H^\circ_{\text{ads}}$ is obtained from the plot of $\ln K_{\text{ads}}$ versus $1/T$ (Fig.3) [19]. As the the experiment proceed at the standard pressure and the solution concentration is not very high close to standard condition, the heat of adsorption can be regarded as the standard heat of adsorption $\Delta H^\circ_{\text{ads}}$ under experimental condition.

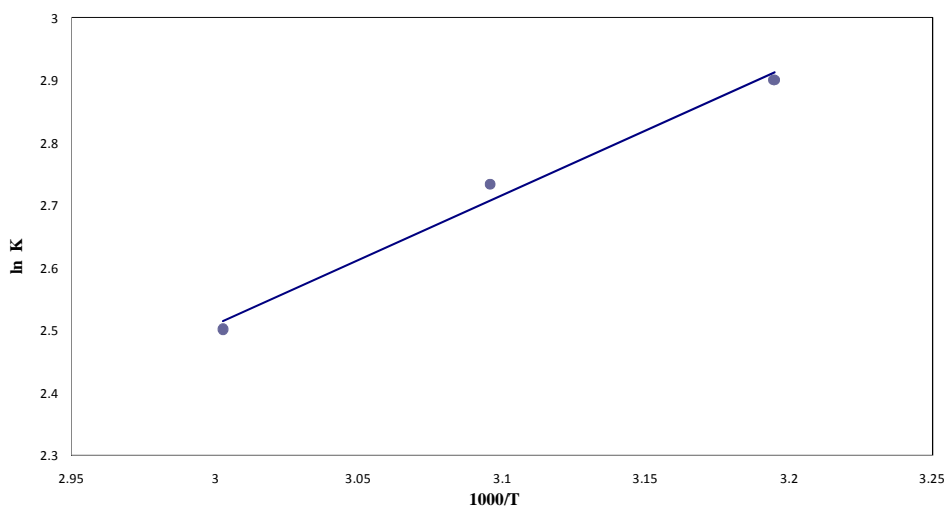


Fig.3. Von't Hoff plot of henna leaves extract in 0.5 M acetic acid

Entropy of adsorption can be calculated from the equation as follow.

$$\Delta G_{\text{ads}} = \Delta H^\circ_{\text{ads}} - T\Delta S^\circ_{\text{ads}} \quad \text{----- (5)}$$

All the thermodynamic parameters wered listed in Table-2.

Table 2. Thermodynamic parameters for the adsorption of henna leaves extract on mild steel surface

Temperature (°C)	K_{ads} kJmol^{-1}	ΔH° kJ mole^{-1}	ΔS° J mole^{-1}	ΔG° kJ mole^{-1}
40	18.18	-17.24	2.43	-17.99
50	15.38		2.74	-18.12
60	12.19		2.43	-18.04

The negative value of ΔH°_{ads} suggest that the process of adsorption of inhibitor on mild steel surface is exothermic. It can be assume that the increase in temperature leads to the increase in desorption of the adsorbed inhibitor molecule from the mild steel surface [21].

The value of ΔS is positive. Generally adsorption is an exothermic process and always accompanied by a decrease of entropy. The reason can be explain as; the process adsorption of inhibitor molecule is accompanied by the desorption of water molecule from the mild steel surface. The adsorption occur as the result of substitution adsorption process between inhibitor molecule present in solution and the water molecule previously adsorbed on the metallic surface[20]. And the thermodynamic values obtained are algebraic sum of the adsorption of inhibitor molecule and desorption of water molecule [21,22].

To investigate the influence of temperature on the corrosion and corrosion inhibition process, the weight-loss measurements were carried out at different temperature 313, 323 and 333 K, in the absence and presence of various concentration of the extract in 1.0 M acetic acid solution for immersion period of 2 h. Result indicates that as the temperature increases the corrosion rate increased and the I.E. decreased (Table.-3). The decreasing trend in the I.E. may be attributed to temperature dependent adsorption/ desorption process influenced in the way that, equilibrium shift towards desorption process, resulting in a decreasing in surface coverage [23]. The value of activation energy (E_a) has been calculated with the help of Arrhenius equation [13].

$$\log \frac{\rho_2}{\rho_1} = \frac{E_a}{2.303R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right) \quad \text{----- (6)}$$

Where, ρ_1 and ρ_2 are the corrosion rate at temperature T_1 and T_2 respectively.

Table 3. Effect of temperature on the corrosion loss (CL), energy of activation (E_a) and heat of adsorption (Q_{ads}) for the corrosion of mild steel in 1.0 M acetic acid containing henna leaves extract as corrosion inhibitor

Inhibitor	Inhibitor Concentration (g/lit)	Temperature						Energy of activation (E_a) kJ mol ⁻¹	Heat of adsorption (Q_{ads}) kJ mol ⁻¹		
		313 K		323 K		333 K			From Arrhenius Polt	313-323 K	323-333 K
		CL mg/dm ²	I.E %	CL mg/dm ²	I.E %	CL mg/dm ²	I.E %	Mean			
Blank		858.37		1430.62		2288.98		42.04	42.47		
Henna	0.1	286.12	66.67	629.47	56.00	1087.27	52.50	57.58	57.90	-38.01	-12.61
	0.25	228.90	73.33	457.80	68.00	858.35	62.50	57.25	57.25	-21.70	-21.75
	0.5	171.67	80.00	343.35	76.00	629.47	72.50	56.24	56.29	-19.65	-16.39
	1.0	114.45	86.67	286.12	80.00	515.02	77.50	64.80	65.25	-40.83	-13.38

Effective area of specimen: 0.2097 dm²

Immersion period: 2 h

It was found that the value of E_a for inhibited system was higher than that of uninhibited system. The higher value of E_a in the inhibited system indicate the inhibitors are more effective at lower temperature [24]. The E_a in inhibited acid was ranging from 57.25 to 64.20 kJ mol⁻¹, which indicates that strong inhibition action of the extract is by increasing the energy barrier for the corrosion process, emphasizing the electrostatic character of the inhibitor (physisorption)[25]. The corrosion rate of mild steel in acetic and the temperature were related by the Arrhenius equation as shown below [26].

$$\rho = A e^{-E_a/RT} \quad \text{-----} (7)$$

Where ρ = corrosion rate, A= constant, R= gas constant, e = base of the natural logarithm, and T = Kelvin temperature.

The value of E_a were also calculated from the slope of the Arrhenius plot of $\log \rho$ versus $1000/T$ (Fig.- 4), shows good agreement with the calculated values.

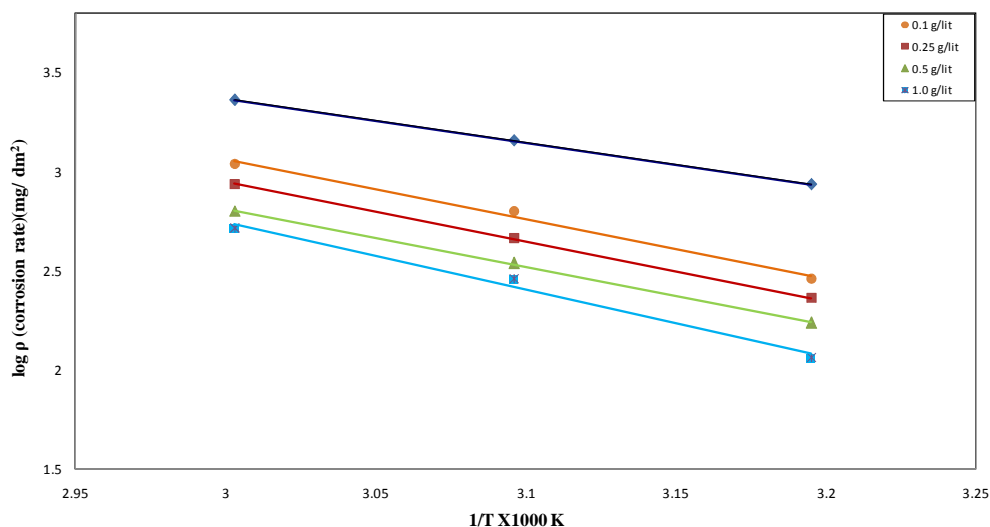


Fig.4. Arhenius plot for corrosion of mild steel in 1.0 M acetic acid solution in absence and presence of henna leaves extract

The values of heat of adsorption (Q_{ads}) were calculated by following equation [14].

$$Q_{ads} = 2.303R \left[\log \left(\frac{\theta_2}{1-\theta_2} \right) - \log \left(\frac{\theta_1}{1-\theta_1} \right) \right] \times \left[\frac{T_1 T_2}{T_2 - T_1} \right] \text{ ----- (8)}$$

Where, θ_1 and θ_2 are the fraction of the metal surface covered by the inhibitors at temperature T_1 and T_2 respectively. The negative and lower values of Q_{ads} supports higher I.E. achieved by spontaneous adsorption of the inhibitor.

2.2. Polarization measurement

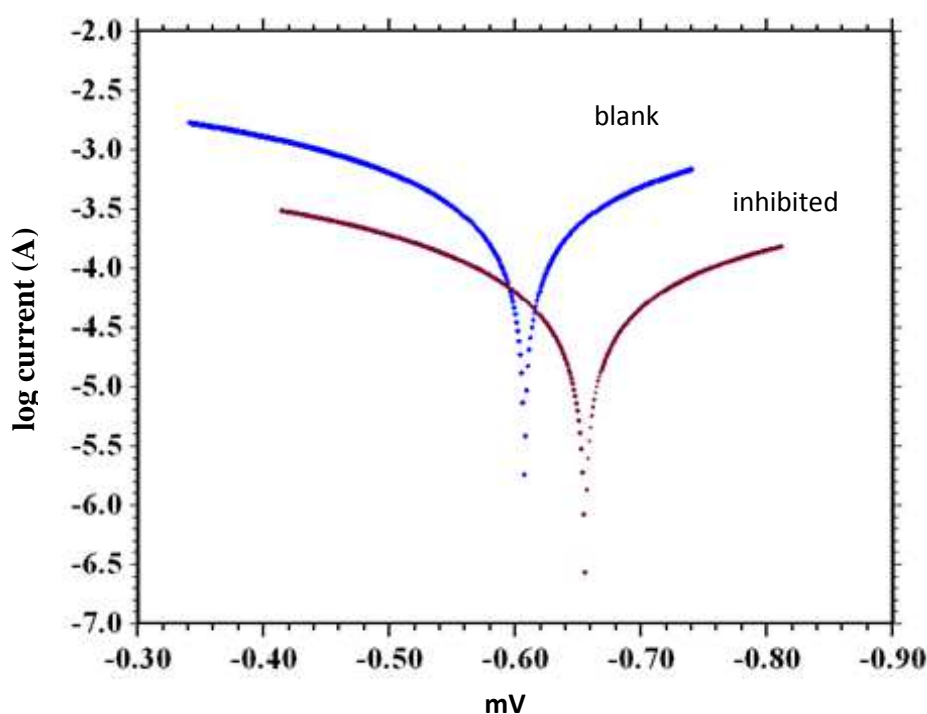
Various parameters like current density (I_{corr}), corrosion potential (E_{corr}) as well as cathodic and anodic Tafel slope (β_c and β_a) were evaluated from the Tafel polarization curve (Fig.-6) and were listed in the Table-5. The corrosion current density (I_{corr}) in case of inhibited system reduce compare to uninhibited system. The IE% was calculated from the I_{corr} as follow.

$$IE \% = \frac{I_{corr,u} - I_{corr,i}}{I_{corr,u}} \times 100 \text{ ----- (9)}$$

Where, $I_{corr,u}$ is corrosion current of uninhibited system and $I_{corr,i}$ is corrosion current of inhibited system.

Table 4. Polarization parameters of mild steel in absence and presence of henna leaves extract in 0.5 M acetic acid solution at 298 K

System	E_{corr} (mV)	Corrosion current density I_{corr} ($\mu\text{A}/\text{cm}^2$)	Tafel Slope (mV/decade)			Inhibition efficiency (IE %) calculated from	
			Anodic $+\beta_a$	Cathodic $-\beta_c$	β (mV)	Weight loss Method	Polarization method
Blank	-0.610	244	5.195	4.659	1.0679		
Henna	-0.665	46.57	5.331	4.649	1.0797	86.27	80.91

**Fig.5.** Polarization curves of mild steel in 0.5M acetic acid blank and inhibited solutions

The value of I_{corr} decreases significantly in presence of inhibitor, this observation indicates that henna leaves extract was effectively inhibit the corrosion of mild steel in acetic acid solution. The presence of henna leaves extract did not change cathodic Tafel slope (β_c) significantly, indicating that inhibitor molecule do not effect the hydrogen evolution reaction. The increase in anodic Tafel slope (β_a) with addition of inhibitor, shows that the adsorbed inhibitor modifies the metal dissolution process[27]. The E_{corr} value of inhibited solution slightly shifted. The change in E_{corr} less than ± 85 mv indicate that the inhibitor is of mixed

type inhibitor[28]. The change in E_{corr} is about 55 mV in presence of inhibitor according to uninhibited solution, reveals that the henna leaves extract act as mixed type inhibitor.

2.3. A C Impedance method

The typical Nyquist plots of mild steel in the absence and presence of henna leaves extract in 0.5 M acetic acid solution was presented in Fig.- 7. From the fig.-7 it was observed that the diameter of the semicircle increases in the case of inhibited acid solution, indicated an increase in corrosion resistance of system.

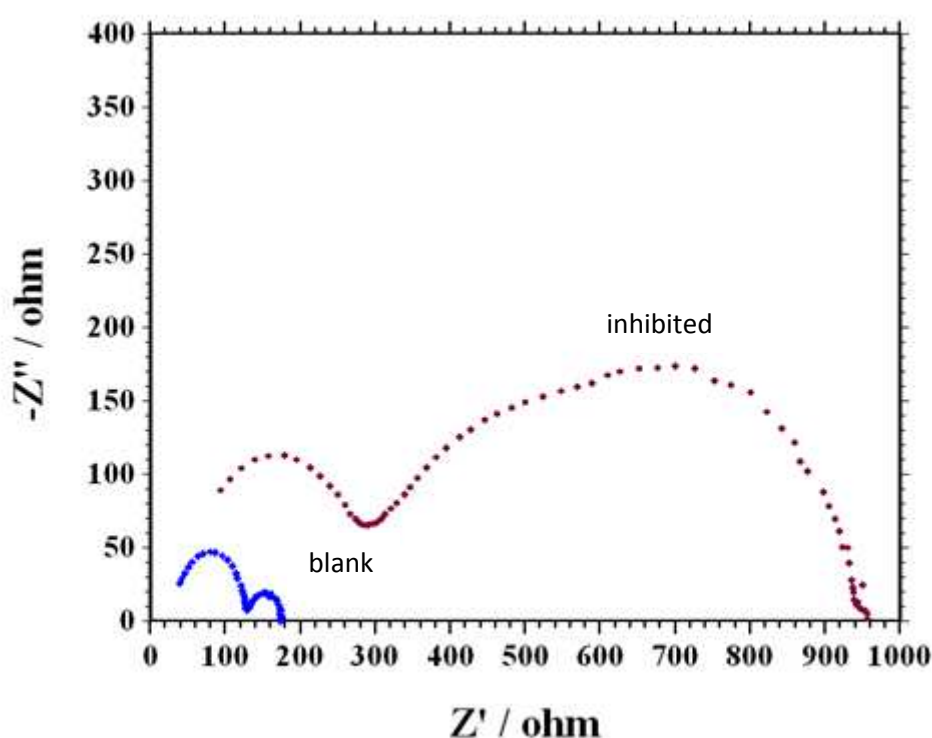


Fig.6. Impedance diagrams obtained for mild steel in 0.5 M acetic acid solution in the absence and presence of henna leaves extract at 1.0 g/lit concentrations

The value of charge transfer resistance (R_{ct}) were calculated from the difference in impedance values at lower and higher frequencies. From the value of charge transfer resistance (R_{ct}) obtained for inhibited and uninhibited acid solution, the value of I.E. of henna leaves extract was calculated from the following equation.

$$IE \% = \frac{R_{\text{ct},i} - R_{\text{ct},u}}{R_{\text{ct},i}} \times 100 \quad \text{----- (11)}$$

The double layer capacitance (C_{dl}) was calculated from the equation as below.

$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}} \quad \text{----- (12)}$$

Where f_{max} is the frequency at which the imaginary component of the impedance is maximum.

Table 5. Impedance parameters for corrosion of mild steel in 0.5 M acetic acid in the absence and presence of henna leaves junipers extract

System	R_{ct} (Ω/ cm^2)	C_{dl} ($\mu\text{F}/ \text{cm}^2$)	Inhibition efficiency (IE %)	
			Impedance measurement	Weight loss method
Blank	56.22	145		
Henna (1.0g/lit)	1399.97	0.6741	95.98	86.27

As the values stated in Table -6, the extract of henna leaves increase the value of R_{ct} . The value of C_{dl} decrease in presence of the inhibitor compare to uninhibited acid solution. This result may be attributed to the adsorption of the component present in the aqueous extract of henna leaves on metal surface [27].

2.3 Mechanism of inhibition:

The result indicates that henna leaves extract performs as good inhibitor for the corrosion of mild steel in acetic acid media. It was reported that henna leaves contain soluble matter Lawson (2-hydroxy-1,4 naphthoquinone) resin and tannin, coumarins, gallic acid and Sterols[29]. Lowson amounts are 1.02% in the leaves [30].The coloring mater is quinine.

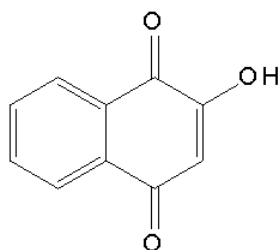


Fig.7. 2- hydroxynaphthalene-1,4-dione

Tannins are found limited inhibition in acidic media than in the alkaline medium [30]. The other constituent of the extract is lawsone, which is present in relatively higher amount. Lawson molecule is a ligand, that can chelate with various metal cations forming complex compounds [31]. Therefore, the formation of insoluble complex compounds, by combination of the metal cations and lawsone molecule adsorbed on the metal surface, it is a probable interpretation of the observed inhibition action of lawsone.

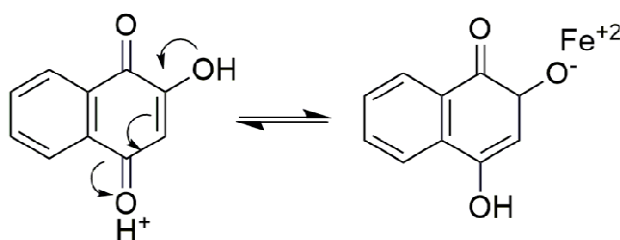


Fig.8.

Aromatic compounds containing delocalized π -electron system in their structure are susceptible to electron delocalization in acidic media, especially a ring containing only carbon (e.g., benzene) [28]. In the case of lawsone molecule, the hydroxyl group contains a pair of electrons, which is delocalized in acetic acid solution that leads to the rearrangement shown in Fig. 8 [32]. The rearrangement occurs as a hydrogen atom migrates with a pair of electrons from an adjacent carbon to the carbon bearing the positive charge. The carbon that loses the migrating group obtains the positive charge. The rearrangement is intramolecular and starting compound and reaction product are structural isomers (Fig.- 8). The metal complexes are stabilized by this rearrangement. The high inhibition efficiencies of the inhibitor in acetic acid for mild steel can be attributed by the formation of these stabilized complexes between lawsone molecules and Fe^{2+} cations present in mild steel in acetic acid media.

3. EXPERIMENTAL

3.1. Method and materials:

Weight loss method

Rectangular specimens of the size $4.5 \times 2.0 \times 0.2$ cm having an area of 0.2097 sq. dm. of mild steel with small hole of 5 mm diameter near the upper edge, were used for the determination of corrosion rate. The specimens were polished by buffing, cleaned with distilled water several times, then degreased by acetone for 1-2 minutes, then dried in warm air by air drier and are preserved in desiccator till use. For weight-loss measurement, one specimen only was suspended by a glass hook, in each beaker containing 230 ml of the test solution and was open

to air at room temperature for 24 h. After the immersion period, the test specimens were clean with distilled water, dried and weighted. Triplicate experiments were performed in each case and the mean value of weight loss data were presented in mg/sq. dm. The concentration of acetic acid studied were taken as 0.5, 1.0, 1.5 and 2.0 M without and with inhibitors having concentration of 1, 0.5, 0.25 and 0.1 g/lit for weight loss measurement at the room temperature of 303 ± 1 K. To study the effect of temperature on corrosion of mild steel, similar experiment was also carried out at various temperatures like 313, 323 and 333 K in 1.0 M acetic acid, using water circulated thermostate with an accuracy of ± 0.5 K, for immersion period of 2 hours. The digital balance of ± 0.001 gm accuracy was used. All the chemical used were of A.R. grade. The corrosive solution was prepared in double distilled water. From the weight-loss data I.E., energy of activation (E_a), heat of adsorption (ΔH_{ads}), free energy of activation (ΔG_{ads}), enthalpy (Q_{ads}) and entropy change (ΔS) were calculated.

Electrochemical measurement

Both the potentiodynamic polarization and electrochemical impedance measurements were carried out using C H instrument (CHI608C). A platinum and Ag/AgCl electrode were used as auxiliary and reference electrode respectively. The test specimen of area 1cm^2 is act as working electrode. All the experiments were carried out at constant temperature 298 K. Initially open circuit potential (OCP) was measured as a function of time, after that the specimen was polarized in cathodic and anodic direction to -200 and +200 mV from the OCP.

Electrochemical impedance spectroscopy (EIS) was carried out in frequency range 1 to 100 kHz and A. C. amplitude of 5.0 mV. The measurement was carried out after stabilization of the electrode at OCP for 30 minute at room temperature.

Preparation of henna leaves extract:

An aqueous extract of henna leaves powder has been used as a corrosion inhibitor. The powder of the crushed henna leaves is extracted for 2 h in boiled water. After filtration, the water was evaporated from the extract. The solid residue left behind was used to prepare various concentrations as the inhibitor.

4. CONCLUSION

1. The corrosion rate of mild steel increases with the increase in acetic acid concentration.
2. The henna leaves extract found to be effective inhibitor for mild steel in acetic acid, and their inhibition efficiency increase with increase in its concentration.

3. The higher values of activation energy indicate that electrostatic interaction between charged inhibitor molecule and the metal surface occurs.
4. Polarization measurement indicates that the henna leaves extract is mixed type inhibitor.
5. The results obtained from weight-loss, potentiodynamic polarization and impedance measurement techniques were in good agreement.

5. ACKNOWLEDGEMENT

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