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Evaluation of Inhibition Effect of *Rosa Damascena Leaves Extract as an Eco-Friendly Inhibitor for Mitigating Corrosion on Mild Steel in 0.5m Sulphuric Acid Medium*

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ABSTRACT

The anti corrosive property of *Rosa damascena* (*RD)* leaves on mild steel in 0.5M sulphuric acid was analysed by mass loss techniques, impedance and polarisation studies. A maximum of 92.92 % inhibition efficiency was reached by using 12v/v % of RD inhibitor. Thermodynamic parameter specifies spontaneous adsorption of the inhibitor on mild steel surface. The adsorption of *Rosa damascena* inhibitor on mild steel surface was found to follow Langmuir adsorption isotherm. Surface analytical techniques validate the formation of protective layer on the inhibitor.

KEYWORDS: Green inhibitor, weight loss, thermodynamic parameters, electrochemical, surface studies

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1. INTRODUCTION

The adverse effect of corrosion towards the environment is a global issue which provokes the investigations in this intensive field of research. Corrosion is a foremost problem which threatens the environment in safety and economic reasons [1,2]. The usage of mild steel in construction, industrial and every day application is enormous because of their excellent mechanical properties and low cost [3]. The destructive nature of acids such as sulphuric and hydrochloric acids tend to deteriorate the mild steel to a greater extent[4]. Literature reveals that the loss produced by corrosion is more than loss due to natural disasters[5]. At the outset the control of corrosion on metallic structures can be triggered by adding active chemical substances to the corrodent. This process is known as inhibition and the substances introduced are called as corrosion inhibitors which is the most emphatic technique to impede corrosion in aggressive medium [6]. The utilisation of synthetic organic inhibitor is the initial report for the corrosion of metals in acid medium, after that enormous number of inhibitors was disclosed as potential compounds for inhibition on mild steel[7]. The toxic and non- eco friendliness of some of the synthetic inhibitors mandates to look forward for eco-friendly, cheap and biodegradable green inhibitors as corrosion mitigators. The anti corrosive property of natural inhibitors may be due to heterocyclic constituents like flavonoids, alkalonoids, polycyclic compounds etc., which enriches the formation of film, thus avrting corrosion. Numerous research works based on the natural materials as corrosion inhibitors are reported in literature. Green materials that have been reported for their inhibitive effect on mild steel in acid medium are *Bombax ceiba*[8] ,*Clerodendrum splendens*[9], *Clivia nobilis*[10] *Mirabilis Jalapa* flowers [11] ,*Tabernaemontana divaricate*[12]etc.,. The present work was carried out to study the inhibition effect of *R.damascena* leaves extract due to their easy availability, cheap and non-toxicity. It comes under Rosaceae family. It can grow upto 2.2 meters with five leaflets. Literature reveals the plant contains terpenes, glycosides, flavonoids, carboxylic acid, quarcetin etc.[13]

2. MATERIALS AND METHODS

2.1. *Preparation of inhibitor*

RD leaves were collected, washed and completely dried. It was powdered and about 1 g of powdered leaves were refluxed in 0.5M sulphuric acid medium for 3 hours at 70^oC to extract the basic ingredients that are soluble in acid. It was then cooled and filtered. The resultant solution was taken as inhibitor and it was refrigerated for further use. From this stock solution, concentrations of 2, 4, 6, 8, 10, 12 v/v% solutions were prepared.

2.2. *Preparation of mild steel specimen*

Mild steel specimen of compositions (0.085 C, 0.368 Mn, 0.128 Si, 0.024 P, 0.026 S, 0.023 Cr, 0.012 Mo, 0.014 Ni and 98.9 % of Fe) was utilised. It is a rectangular specimen of dimensions 3 cm x 1 cm x 0.1 cm with a tiny hole in the top of the specimen. After removing impurities, it is abraded using emery, degreased with acetone, dried and desiccated.

2.3. *Weight loss technique*

Corrosion rate is usually evaluated by weight loss technique. The polished and weighed metal specimens were immersed in 100 ml of 0.5M H_2SO_4. Similar to blank, RD inhibitor of different concentrations (2, 4, 6, 8. 10, 12v/v %) in 0.5M H_2SO_4 were carried out. The specimens were retrieved after 3 hours from the solution, washed with water, dried and reweighed to evaluate weight loss. From the initial and final weight of the specimen corrosion rate, surface coverage and inhibition efficiency was determined using the formula,

$$\text{Inhibition efficiency } (\%\text{ IE}) = \frac{W_{bn} - W_{in}}{W_{bn}} \text{x}100 \quad (1)$$

$$\text{Corrosion rate } (C_r) = \frac{534 \text{ x weightloss(g)}}{\text{Density}(g/cm^3) \text{ x Area(cm) x Time(hr)}} \quad (2)$$

where W_{bn} – weight loss without inhibitor, W_{in}-weight loss with inhibitor. Similar procedure was followed to study the influence of temperature on the inhibitory properties of RD inhibitor in the temperature range (313-333 K) and activation, the rmodynamic parameters were calculated using the formula,

$$\log CR = \frac{-E_a}{2.303RT} + \log A \quad (3)$$

$$\text{Corrosion rate} = \frac{RT}{Nh} \exp\left(\frac{\Delta S^o}{R}\right) \exp\left(\frac{-\Delta H^o}{RT}\right) \quad (4)$$

where E_a-apparent activation energy, A-frequency factor, ΔS^o - entropy of activation, ΔH^o - enthalpy of activation, N - Avogadro number, T - absolute temperature and R - universal gas constant.

2.4. *Electrochemical and Impedance measurements*

In electrochemical measurements, Nyquist plots obtained from impedance measurements and Tafel plots from polarisation datas. IVIUM compactstat Potentiostat/Galvanostat electrochemical analyser utilised for this purpose. Impedance measurements were carried out in a frequency range of

10 kHz to 0.01 Hz. From this measurement, charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) were determined. Polarisation measurements were carried out in the range of -200 mV to +200mV

$$\text{Inhibition efficiency (\%IE)} = \frac{R_{ct(in)} - R_{ct(bk)}}{R_{ct(in)}} x100 \quad (5)$$

$$\text{Inhibition efficiency (\%IE)} = \frac{I_{corr(bk)} - I_{corr(in)}}{I_{corr(bk)}} x100 \quad (6)$$

2.5. *Surface analysis*

2.5.1. SEM analysis

The working electrodes (MS) were immersed in control/ inhibitor of optimum concentration of 12v/v % of RD for 3 hours. After the specified time the sample coupons were retrieved from the solution, washed with water and air dried. The coupons were investigated for their surface morphology using biomedical research microscope (Mumbai, India).

2.5.2. *Atomic Force Microscopy*

AFM is a powerful tool to analyse the surface morphology of the metal specimen. For this study working electrodes (MS) were suspended in the test solution in the uninhibited and inhibited solution of RD inhibitor for a time of 3 hours. The specimens were removed, washed and dried and evaluated for the surface studies using a multimode scanning probe microscope (NTMDT, NTEGRA prima, Russia).

3. RESULTS AND DISCUSSION

3.1. *Weight loss technique*

The impact of RD extract on resisting corrosion has been evaluated by weight loss measurements[14]. It has been noticed from Table 1 that by varying the concentration of RD inhibitor the inhibition efficiency increases and attained a maximum efficiency of 92.92 % at 12v/v %.

Table 1: Inhibition efficiency of mild steel in 0.5M H_2SO_4 in the absence and presence of different concentrations of *R. damascena* leaves extract

Name of the Inhibitor	Conc. (v/v %)	IE (%)	Surface Coverage(θ)	CR (mpy)
Blank	-	-	-	7682.246
RD	2	78.00	0.7800	1689.66
	4	84.91	0.8491	1159.11
	6	88.06	0.8806	917.24
	8	89.56	0.8956	802.16
	10	91.27	0.9127	670.50
	12	92.92	0.9292	543.71
	14	92.98	0.9298	543.45

It is clear from the results that even at minimum concentration of 2v/v % the efficiency is 78 %. These results are evident of the anti corrosive property of RD inhibitor in 0.5M sulphuric acid. The inhibition efficiency increases as the concentration of RD inhibitor increased from (2v/v % - 12v/v %) which specifies the dependency of the inhibition effect on concentration. This behaviour may be attributed to the increase in surface coverage (θ) by the active species in the extract on the mild steel specimen at higher concentration leading to the formation of film on the surface[15]. The weight loss measurements provide corrosion rate, surface coverage and inhibition efficiency of RD inhibitor and it is presented in Table 1.The variation of inhibition efficiency with different concentrations is depicted in Fig. 1.

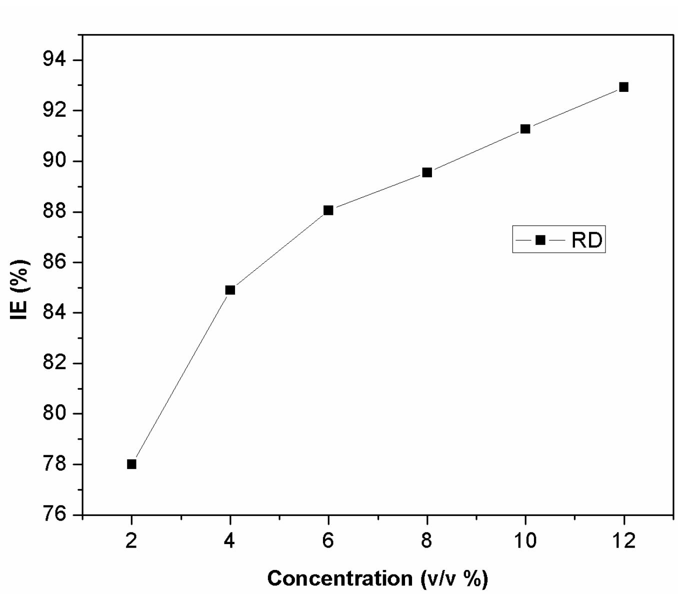


Fig. 1 Variation of inhibition efficiency with concentration on mild steel in 0.5M H_2SO_4 in presence of *R. damascena*

3.2 Effect of Temperature

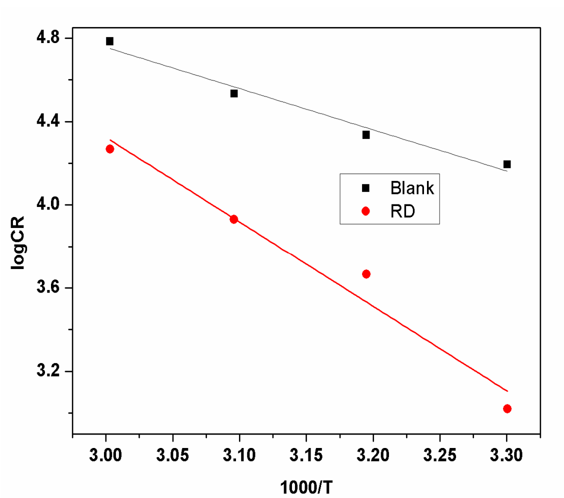
Inhibitor	Conc. (v/v %)	Temperature							
		303		313		323		333	
		IE%	CR(mpy)	IE%	CR(mpy)	IE%	CR(mpy)	IE%	CR(mpy)
Blank	-	-	7705.17	-	10666.10	-	16822.04	-	29997.04
RD	2	77.20	584.19	55.08	4791.04	43.47	9508.94	39.94	18017.24
	6	87.53	319.40	77.40	2410.88	68.69	5266.49	54.89	13531.66
	12	91.99	205.29	78.69	2273.37	79.65	3423.22	69.64	9106.64

Table 2. Variation of inhibition efficiency with concentrations of *R.damascena* inhibitor at different temperatures

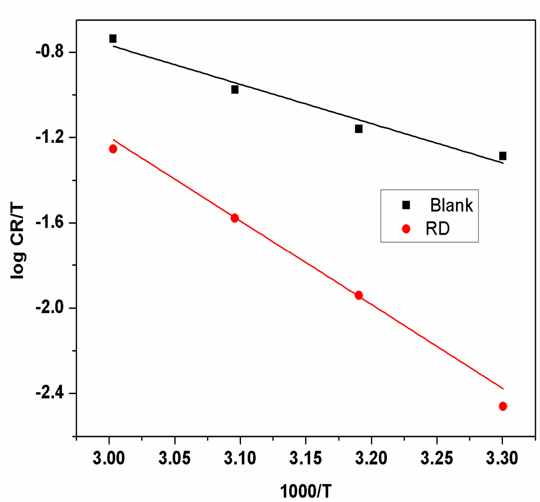
To evaluate kinetic and mechanistic features of corrosion on mild steel surface without and with the addition of inhibitors temperature studies were carried out at a range of 313-333K. The inhibition efficiency presented in Table 2 indicates that as temperature increases the inhibition efficiency decreases with maximum inhibition efficiency of 69.64 % at 333K. It may be due to adsorption and desorption process. It is well known that adsorption and desorption process by the inhibitor takes place at the surface of the metal but at a particular temperature equilibrium occurs between these processes and as the temperature increases the equilibrium between the two processes are shifted which accounts for the higher desorption rate at higher temperatures[16].

3.3. *Kinetics and Thermodynamic parameters*

The influence of temperature is related to equilibrium and kinetics[17]. Arrhenius equation 3 is related to corrosion rate to temperature. Arrhenius and transition plots are shown in Figs. 2a and 2b. A straight line plot was attained for RD inhibitor.



(a)



(b)

Fig. 2 (a) Arrhenius plot (b) Transition plot of corrosion on mild steel with and without the addition of inhibitor

The slope of the linear plot provides the value of E_a. ΔH^o and ΔS^o can be computed from the slope ($-\Delta H^o/ 2.303R$) and intercept [$\log(R/Nh) + \Delta S^o/(2.303R)$]. Table 3 shows the data of E_a. It is inferred that higher E_a value (85.93 kJ mol^{-1}**)** in presence of RD inhibitor than blank (37.87 kJ mol^{-1}) implies corrosion is resisted with the formation of inhibitor film barrier. The positive value of ΔH^o for RD inhibitor (57.18 kJ mol^{-1}) denotes endothermic nature implying that metal dissolution is difficult[18]. ΔS^o is negative reflecting the decrease in disorderness[19]

Table 3. Activation parameters for mild steel corrosion in 0.5M H_2SO_4 in the uninhibited and inhibited solutions

Inhibitor	E_a kJ mol^{-1}	ΔH^o kJ mol^{-1}	ΔS^o J $mol^{-1}K^{-1}$	$+\Delta G^o$ kJ mol^{-1}			
				303	**313**	**323**	**333**
Blank	**37.87**	35.24	-46.52	12.31	13.37	14.4	15.5
RD	**85.93**	57.18	-19.57	5.55	5.93	6.11	6.30

The calculated ΔG^o value is positive which confirms that activated complex is unstable. Figs. 3a and 3b represents VantHoff and Gibbs Helmholtz plots with slope (ΔH^o_{ads} / R) and intercept [$(\Delta S^o_{ads}/R)$ +ln (1/55.5)]. The negative value of ΔH^o in Table 4 indicates exothermic adsorption process [20].

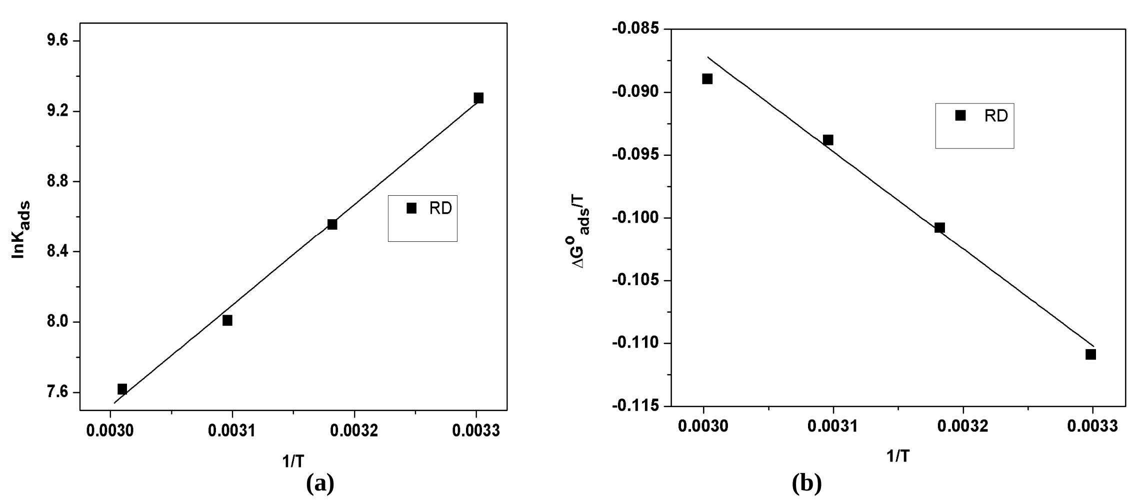


Fig. 3 (a) Vant Hoff plots (b) Gibbs Helmholtz plots of corrosion on mild steel in presence of RD inhibitor

Table 4. Thermodynamic adsorption parameters from Van'tHoff and Gibbs-Helmholtz equation for metal sample in 0.5M H_2SO_4 containing inhibitor

Name of the inhibitor	Van'tHoff equation		Gibbs-Helmholtz equation	ΔG°_{ads} kJ mol^{-1}			
	ΔH°_{ads} kJ mol^{-1}	ΔS°_{ads} kJ mol^{-1}	ΔH°_{ads} kJ mol^{-1}	303 K	313 K	323 K	333 K
RD	-28.22	-0.0366	-28.22	-28.73	-29.11	-30.79	-32.54

3.4. *Adsorption Isotherm*

Based on the degree of adsorption of the active inhibitor molecules on the metal surface the inhibition efficiency can be calculated [21]. It is understood that the efficacy of the inhibitor and their stability of the protective layer is related to the physical or chemical adsorption. The adsorption isotherms are useful in understanding the adsorption process and their mechanisms. Weight loss measurements provide data for concentration(C) and surface coverage (θ) and were utilised for plotting adsorption isotherms like Freundlich, Langmuir, Temkin and Frunkin isotherms. However the current data fits well in Langmuir adsorption isotherm. It can be elaborated based on the relationship between concentration of the inhibitor and surface coverage by an equation,

$$\frac{C_{in}}{\theta} = \frac{1}{K_{ads}} + C_{in} \qquad (6)$$

where K_{ads} represents equilibrium constant for adsorption process.

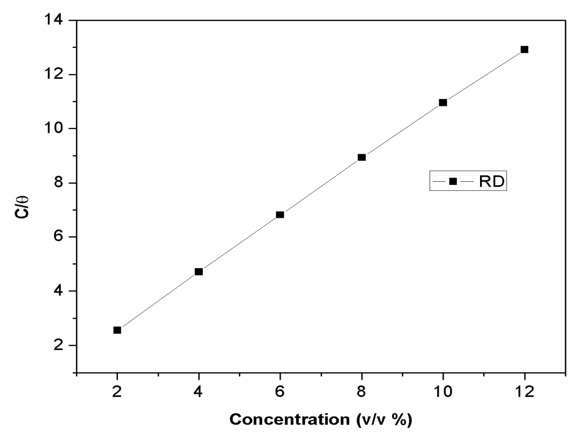


Fig. 4 Linear fitting of *R.damascena* leaves extract to Langmuir adsorption at different concentrations.

In Fig. 4 the linear plot with the regression coefficient (R^2) unity specifies that the RD inhibitor adsorbed on the metal surface obeys Langmuir adsorption isotherm. This fact implies that the mode of adsorption of the inhibitor on the metal surface is physisorption predominantly [22]. The value of K_{ads} is acquired from the intercept of the linear plot and it is tabulated in Table 5.

Table 5. Langmuir isotherm parameters for adsorption of *R.damascena* extract on mild steel in 0.5M H_2SO_4.

Name of the inhibitor	R^2	Slope	K mol lt^{-1}	$-\Delta G^{\circ}_{ads}$ kJ mol^{-1}
RD	0.9996	1.03	554	26.39

Higher value of K_{ads} denotes a vibrant adsorption of the active species in the inhibitor on mild steel which in turn implies the effective inhibition activity .The value for ΔG^o_{ads} can be computed by using the relation,

$$\Delta G^{o}_{ads} = -RT\ln(55.5K) \quad (7)$$

The value of ΔG^o_{ads} for RD inhibitor is -26.39 kJ/mol specifies physisorption and negative value implies spontaneity of the adsorption behaviour as well as stability of the film on the metal surface [23].

3.5. *Electrochemical Impedance Spectroscopy*

The impedance studies have been carried out in corrodent medium at room temperature without and with different concentrations of RD inhibitor. The difference in impedance at two frequencies (lower and higher) provides charge transfer resistance. It is clear from Fig. 5 that semicircle shaped Nyquist plots increases as concentration of RD inhibitor increases. Single semicircle in Nyquist plot related to one capacitive loop[24]. Nyquist plot clearly validates that the impedance response of mild steel in corrodent medium without inhibitor has expressively changed with different concentrations of the inhibitor.

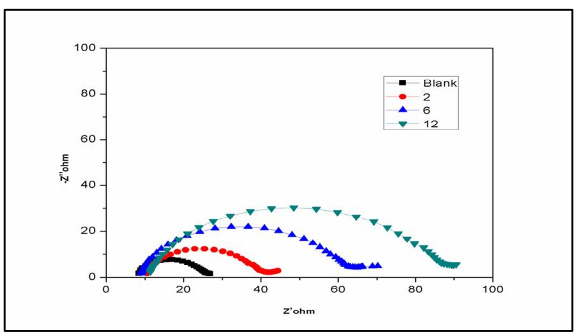


Fig. 5 Nyquist plot of mild steel in 0.5M H_2SO_4 solution without and with different concentrations of *R.damascena* inhibitor

Table 6 indicates an increased value of R_{ct} with increase in the concentration of the inhibitor which in turn is related to inhibition efficiency. Decrease in C_{dl} with addition of the inhibitor contributes for the increased value of inhibition efficiency. Due to reduction in local dielectric constant/ increase in thickness of the electrical double layer C_{dl} value decreases, suggesting that active phyto constituents in the RD inhibitor acts by adsorption at the metal/electrolyte interface. The impedance study also validates that RD inhibitor acts as a good corrosion inhibitor in 0.5M H_2SO_4.

Table 6. Data from the impedance spectroscopy analysis for metal specimen without and with different concentrations of inhibitor in 0.5M H_2SO_4.

Name of the inhibitor	Conc. (v/v %)	R_{ct} (ohm cm^2)	C_{dl} (μF/cm^2)	IE (%)
Blank	-	17	61	-
RD	2	45	55.8	62.2
	6	70.3	43.4	71
	12	104.2	38.2	80.7

Bode plots for mild steel corrosion without and with the addition of inhibitor at varying concentrations (2, 6 and 12v/v %) in 0.5M H_2SO_4 are illustrated in Fig. 6. It is interpreted from the Bode plot that the semicircle in the Nyquist plot is related to a single time constant. The increase in resistance with the addition of the inhibitor is associated with the adsorption of the active components by replacement of water molecules.

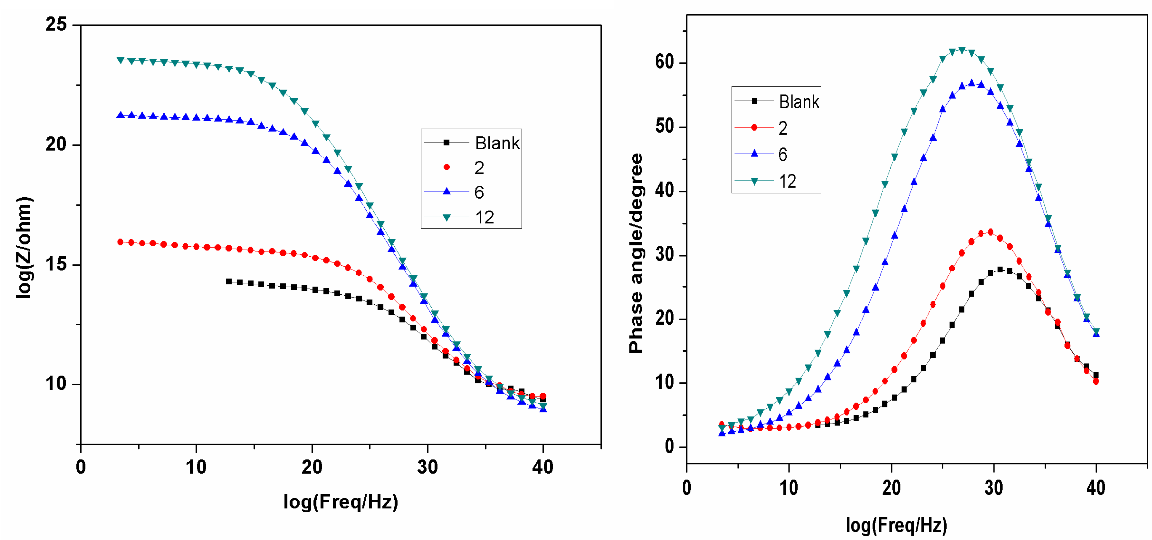


Fig. 6 Bode plot of mild steel in 0.5M H_2SO_4 solution in the absence and presence of different concentrations of *R. damascene* inhibitor

3.6. *Potentiodynamic Polarisation Studies*

Anodic and cathodic polarisation curves for mild steel in 0.5M H_2SO_4 in uninhibited and inhibited solutions of RD inhibitor is presented in Fig.7. RD inhibitor can impede anodic dissolution and cathodic hydrogen liberation reaction[25]. Various parameters such as I_{corr} (corrosion current density) , E_{corr} (corrosion potential) ,anodic (b_a) and cathodic (b_c) slopes are tabulated in Table 7. It can be noted that with increasing concentration of RD inhibitor current density decreases. It means that the adsorption of inhibitor on mild steel takes place thus retarding corrosion. Tafel plots indicates that the presence of inhibitor has influenced both anodic and cathodic half reaction.

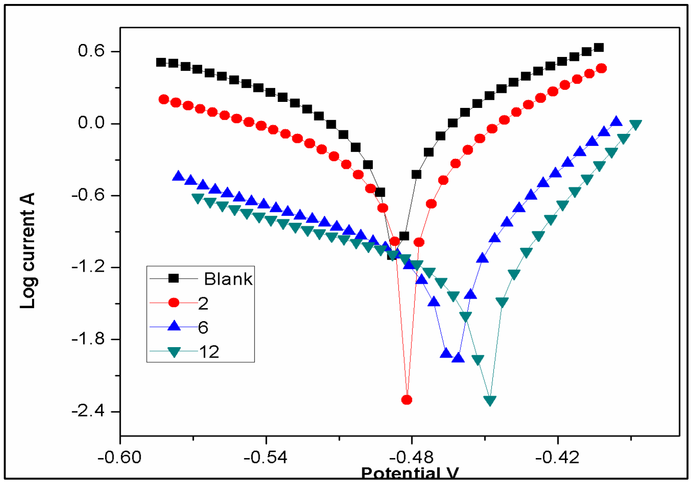


Fig. 7 Polarisation curves showing influence of increasing concentrations of *R. damascena* leaves extract on mild steel in 0.5 M H_2SO_4

Shift in E_{corr} is 45mV in the presence of inhibitor in comparison with blank solution implies *Rosa damascena* leaves act as mixed type inhibitor[26]. A change in anodic and cathodic slopes also supports the mixed nature of the inhibitor.

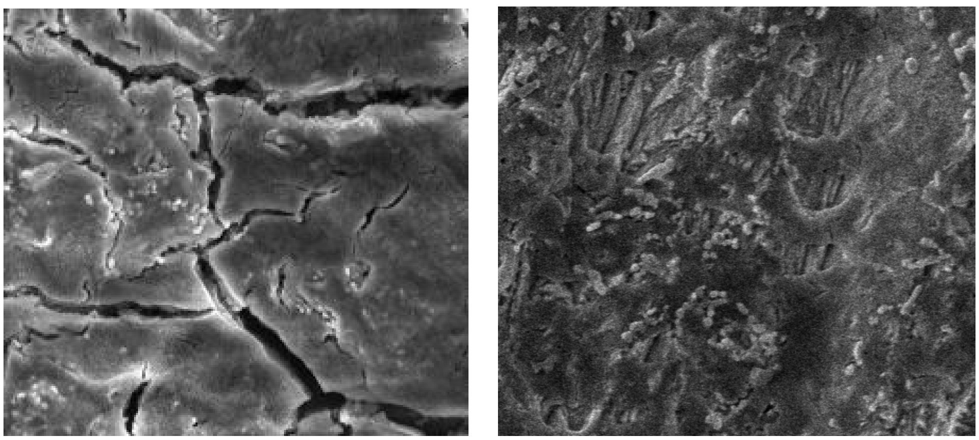
Table 7. Polarisation curves for metal specimen in the absence and presence of different concentrations of *R.damascena* in 0.5M H_2SO_4.

Name of the inhibitor	Conc. (v/v %)	Tafel slopes (mV/dec)		$-E_{corr}$ (mV)	I_{corr} (μA/cm^2)	IE (%)
		b_a	b_c			
Blank	-	62	91	490	520	-
	2	77	148	478.2	210	60
	6	51	160	460	161	69
RD	12	43	180	445.2	112	78.5

3.7. *Surface Studies*

3.7.1. SEM analysis

The mild steel specimens suspended in corrodent medium (0.5M H_2SO_4) and inhibited corrodent medium (0.5M H_2SO_4 + 12v/v % RD) for 3 hours. After 3 hours, the working electrode (MS) was removed, washed, dried and SEM analysis were carried out. It can be viewed from Fig. 8a that in aggressive medium the metal specimens are highly damaged with large cracks and pits indicating that corrosion degraded the metal specimen. In contrast to that the image of steel surface in presence of inhibitor in Fig. 8b is better than the blank indicating a protective layer is formed by adsorption of the inhibitor on the metal surface thus preventing corrosion. The images confirm that RD inhibitor is effective in controlling corrosion by hindering the reactive active sites on the mild steel surface.

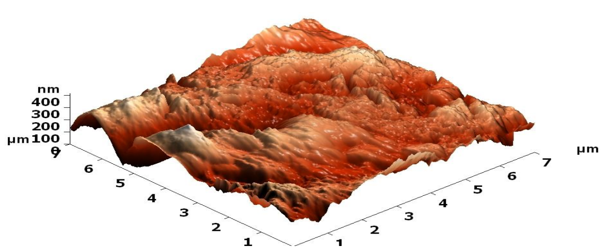


(a) (b)

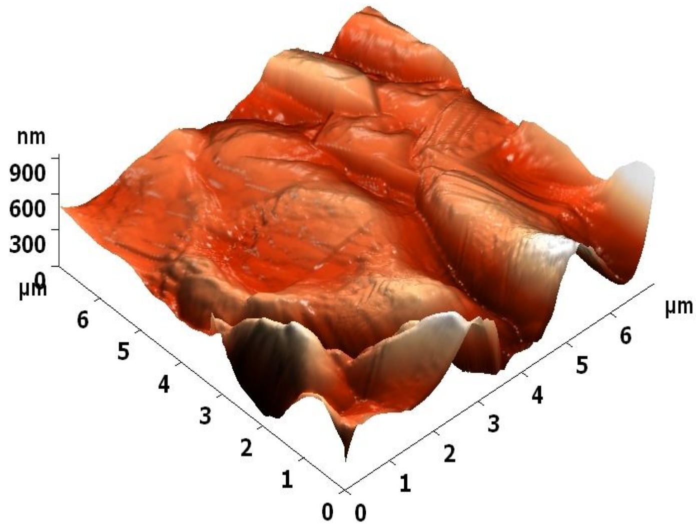
Fig. 8 SEM images of mild steel specimen (a) 0.5M H_2SO_4 treated mild steel (b) 0.5M H_2SO_4 + 12v/v % inhibitor treated mild steel.

3.7.2. Atomic Force Microscopy

AFM images of mild steel specimen immersed in 0.5M H_2SO_4 and in presence of RD inhibitor at 12v/v % are depicted in Figs.9 a and b. Rough surface was more for blank specimen than inhibited specimen. Average roughness for blank specimen is 180.17 nm and for RD inhibitor it is 89.26 nm. These results also validate the formation of protective layer on the metal surface [27,28].



(a)



(b)

Fig. 9 AFM images of mild steel specimen (a) 0.5M H_2SO_4 treated mild steel (b) 0.5M H_2SO_4 + 12v/v % inhibitor treated mild steel.

3.8. *Mechanism of Inhibition*

The inhibitory action of the inhibitor on mild steel specimen may be assumed that the organic molecule in the inhibitor adsorbs at the mild steel /interface in a corrodent medium[29]. The efficiency of the inhibitor depends on adsorption sites, mode of interaction, charge density, molecular weight[30]. In literature, it has been reported that *Rosa damascena* extract contains terpenes, flavonoids, quarcetin etc., It is known that these active organic molecules are protonated in aqueous acid solution. The adsorption of these organic molecules on the mild steel surface may happen in one or combinations of the following

(a) An interaction occurs between the π-electrons of the organic molecule in RD inhibitor and vacant d orbital of metal.

(b) The metal acquires negative charge; hence adsorption occurs through electrostatic interaction between the protonated molecules and metal surface.

RD inhibitor is an effective inhibitor due to the combined effect between the adsorbed SO_4^{2-} ions and protonated active species of the inhibitor. Thus organic molecules in RD inhibitor form a barrier between the corrodent medium and the mild steel surface through adsorption process thus protecting the metal from corrosion.

CONCLUSION

- In the current investigation, the inhibitive performance of acid extract of *Rosa damascena* leaves against corrosion on mild steel in 0.5M H_2SO_4 was investigated.
- An inhibition efficiency of 92.92 % was attained at 12v/v % at room temperature.
- Adsorption of RD inhibitor followed Langmuir adsorption isotherm.
- Kinetic and thermodynamic parameters were calculated.
- Polarisation studies implied that RD inhibitor affects both anodic and cathodic reactions thus acting as a mixed type inhibitor.
- Surface studies of RD inhibitor revealed the formation of protective barrier.

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