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Research paper

Corrosion Inhibition of Morus Rubra Leaf Extract on Mild Steel in Acidic Media

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Abstract

The inhibition effect of Morus rubra leaf extract on the corrosion of mild steel in acidic medium has been investigated by using Galvanostatic polarization, electrochemical impedance spectroscopy and surface analysis by SEM (Scanning Electron Microscopy) and AFM (Atomic Force Microscopy). Corrosion inhibition efficiency increases with increase in plant extract concentration. Scanning Electron Microscopic studies and Atomic Force Microscopic studies also investigate the change in surface morphology by using inhibitor. The result obtained shows that plant extract plays a significant role in corrosion inhibition and serve as an effective inhibitor for the corrosion of mild steel in sulphuric acid medium. Electrochemical methods and surface morphology are widely used for the evaluation of the efficiency of corrosion inhibitors.

Key Words: Plant extract, Corrosion inhibitor, Galvanostatic Polarization, Electrochemical impedance spectroscopy, Scanning electron microscopy, Atomic force microscopy

1. Introduction

Mild steel is used as constructional material in large number of industries due to its low cost and excellent mechanical properties [Behpour et al., 2011]. Acid solutions are widely used in industries for pickling, acid cleanings [Schmitt et al., 1984]. Because of aggressiveness of acids, inhibitors are generally used to inhibit the corrosion rate of metal surfaces. There are so many efficient inhibitors that are used to reduce corrosion of mild steel in acidic medium. Recent research have shown that these green inhibitors exhibit good inhibition efficiency and serve as most effective eco-friendly corrosion inhibitors as compared to chemical inhibitors.. Morus rubra, commonly known as the red Mulberry, is a species of Mulberry native to eastern and central North America. Several workers have reported the effect of synthetic inhibitors on the corrosion of mild steel in acidic media. The synthetic inhibitors exhibit good inhibition efficiency but they are too expensive to synthesize, toxic in nature and have hazardous effect on environment [Raja and Sethuraman (2008)],[Ec Hosary et al., (1972)] and they pollute environment during synthesis. This lead to the use of some natural products which are readily available, non toxic to environment and act as corrosion inhibitors [Satapathy et al., (2009)]. The extract of plant contain numerous naturally environmental organic compounds which contain nitrogen, sulphur and oxygen atoms and these atoms provides major adsorption centers[Ostovari et al., (2009)]. In addition to this, they also contain aromatic rings double and triple bonds, all these together increase the adsorption process and results in the formation of protective layer on the surface of metal [Muthukrishnan et al., (2013), Popova et al., (2004)]. The inhibition abilities of plant extracts depend upon the parts of the plant used [El-Etre (2007)]. Leaf extract and fruit extract of same plant have varying degree of inhibition abilities. Some inhibitors are effective in one medium may not be much effective in another medium.[Saleh et al., (1983)] worked over the aqueous extract of some plant leaves and fruit peels on corrosion of mild steel in acidic medium.

The aim of present work is to study the inhibitive action of Morus rubra leaf extracts on corrosion behavior of mild steel in acidic medium through polarization measurement and electrochemical impedance spectroscopy methods. Through Temperature kinetic studies, Different thermodynamic parameters can be calculated. These parameters explain the mode of adsorption and inhibited metallic surfaces are examined with Scanning electron microscopy and Atomic force microscopy studies.

2. Experimental

2.1. Preparation of Morus rubra leaves extract

Morus rubra leaves were dried in an oven and crushed. The powered leaves were refluxed with 0.5M sulphuric acid for three hours.
The extracted solution was then filtered and filtrate used as stock solution. The testing solutions of different concentration were prepared from stock solution. The different concentrations were 10%, 20%, 30%, 40%.

2.2. Specimen preparation

Mild steel specimens have 1.92% C, 0.165% Si, 0.60% Mn, 0.203% Cu, 0.034% S and 95.5% Fe encapsulated in an Araldite with exposed area of 1cm2 was used as working electrode for potentiodynamic polarization measurement and EIS measurements. The surface area was abraded successively with 220, 400, 800 and 1000 grades of emery papers to obtain uniform surface degreased with acetone and washed with distilled water before each electrochemical experiment [Oguzie. (2008)].

2.3. Electrochemical measurements

Electrochemical studies were carried out by using electrochemical work station impedance analyzer model CHI 760D. A cell assembly usually contains three electrodes. Mild steel specimen as working electrode, platinum electrode as the counter electrode and saturated calomel electrode as the reference electrode. Before carrying out the electrochemical measurement, a stabilization period of 30 min was allowed to attain a stable value of open circuit potential(OCP). Potentiodynamic polarization curves were recorded at a scan rate of 0.01mVs-1 by adding -0.9 and +0.0V to the open circuit value. Electrochemical impedance spectroscopy measurements were carried out using alternating current (AC) signal with amplitude of 5mV at OCP in the frequency range from 105 Hz to 1Hz.

2.4. Surface analysis

Surface of mild steel was abraded by emery papers of different grades to obtain uniform surface, degreased with acetone washed with distilled water before the experiment. Polished coupons were subjected to corrosion by dipping them in 0.5 M H2SO4 solution in the absence and presence of highest and lowest concentration of inhibitor for 24 hours. Then Surface morphology was performed using Ziess scanning electron microscopy and Naio AFM Nanosurf.

3. Result and discussion

3.1. Potentiodynamic Polarization

Potentiodynamic anodic and cathodic polarization curves for mild steel in presence and absence of inhibitor are shown in Fig 1. The respective corrosion current parameters including corrosion potential (Ecorr), corrosion current density (Icorr), cathodic slope (bc), anodic slope(ba) and percentage of inhibition efficiency calculated from table 1. Inhibition efficiency can be calculated as

IE% = (i0corr - icorr / i0corr) × 100

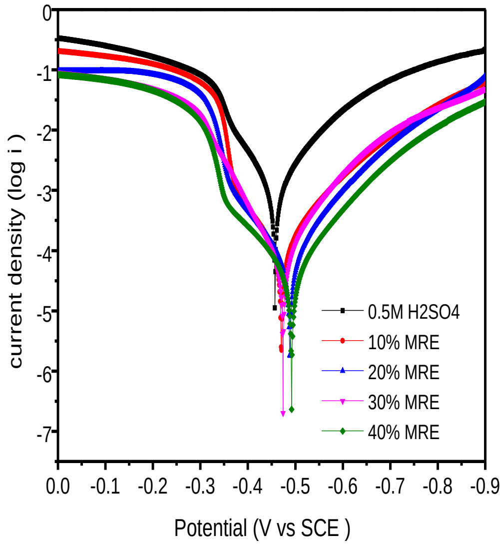
Where i0corr and icorr are corrosion current density in absence and in presence of inhibitor

Table 1: Potentiodynamic polarization parameters for the corrosion of mild steel in 0.5 M H_2SO_4 having different concentrations of MRE(Morus rubra Extract).

Temp.	**conc.**	**$-E_{corr(mV)}$**	**$b_{c(Mv/dec)}$**	**$b_{a(mV/dec)}$**	**$I_{corr(mA/cm^2)}$**	**% IE**
	40%	492	124	104	0.0494	95%
298K	30%	474	98	66	0.0733	93%
	20%	489	105	95	0.0788	92%
	10%	471	115	45	0.0809	91%
	$0.5MH_2SO_4$	457	114	61	1.017	-
	40%	506	164	110	0.1179	91%
308K	30%	529	174	114	0.1517	88%
	20%	502	163	116	0.2064	83%
	10%	513	197	121	0.2173	82.8%
	$0.5MH_2SO_4$	429	116	61	1.267	-
	40%	509	176	112	0.2010	89.82%
318K	30%	527	178	105	0.2962	85%
	20%	523	170	115	0.3382	83%
	10%	492	174	126	0.3811	81%
	$0.5MH_2SO_4$	474	144	66	1.984	-
	40%	518	207	116	0.3632	88%
328K	30%	480	105	92	0.4807	84%
	20%	520	179	120	0.5190	82.7%
	10%	505	170	117	0.5875	80%
	$0.5MH_2SO_4$	470	133	63	3.009	

It was evident from the Table 1 that the corrosion current value (Icorr) decreases with the increase in concentration of inhibitor. No definite trend was observed in the shift of Ecorr values. Both anodic and cathodic branches modified with the addition of inhibitor. Corrosion was inhibited with the increase in MRE concentration and inhibition efficiency was improved. At 10%, IE was 91% and as the concentration increased to 40%, IE was 95%. Temperature influenced the corrosion rate in the presence of MRE. Inhibition efficiency decreased with increase in temperature. At 40% inhibitor concentration,, inhibition efficiency reduced from 95% at 298 K to 88% at 328 K.

At a particular concentration, the decrease in corrosion current value is less at higher temperature thus inhibition efficiency also decreases. According to a literature report when corrosion potential is more than ± 85 mV with respect to that of the blank, the inhibitor can be considered distinctively as cathodic or anodic type. However, for MRE as inhibitor of corrosion for mild steel, the change in Ecorr values is less than ± 85 mV suggesting that MRE act as a mild type of inhibitor [Petkov and Manolov, (1978), Muthukrishnan et al.,(2013)]. Both anodic and cathodic polarizations are influenced simultaneously, with a slightly more shift towards cathodic direction.



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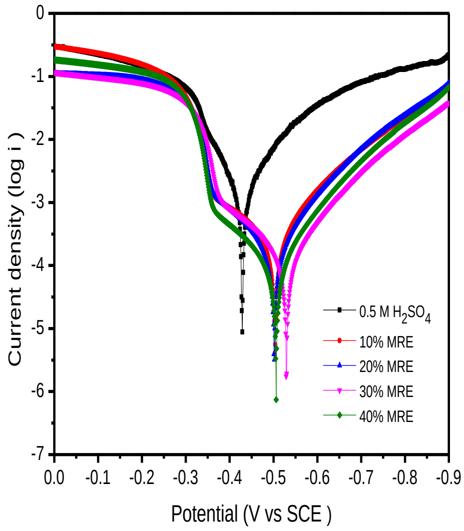

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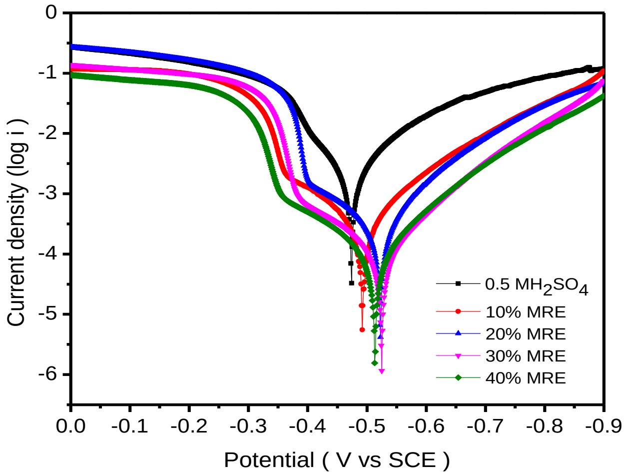

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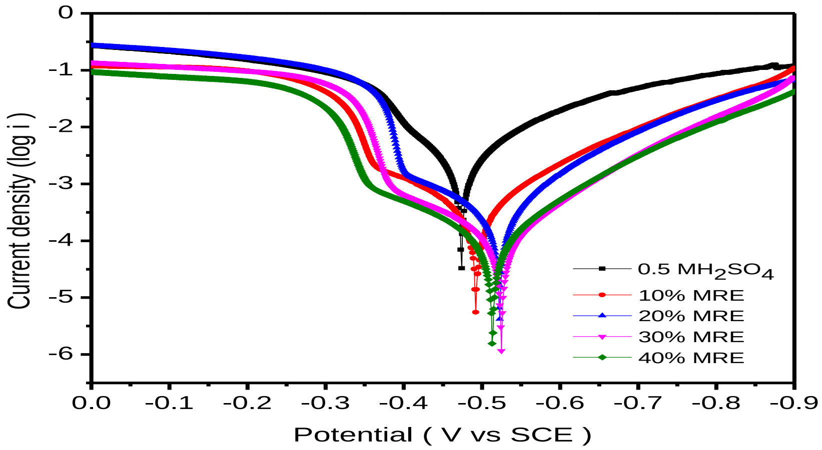


1 (d)

Figure 1: Polarization curves for mild steel in 0.5M H2SO4 with different concentrations of MRE (a) 298K (b) 308K (c) 318K (d) 328K

The change in the value of bc and shift in the anodic Tafel slope ba values suggest that MRE extract act as corrosion inhibitor suppressing both anodic and cathodic reactions by getting adsorbed on the mild steel surface. These results showed that MRE extract act as mixed type of inhibitor.

3.2. Electrochemical impedance spectroscopy

Impedance diagrams for mild steel in 0.5 M H2SO4 with and without different concentration of MRE are given in Fig. 2 and impedance parameters such as Rct , Cdl and fmax obtained from electrochemical impedance measurements are shown in Table 2. Impedance diagrams show depressed semicircles. Each spectrum consists of a large capacitive loop in high frequency and an inductive loop in low frequency values [Singh et al., (2016)]. Diameter of capacitive loop increases with increase in inhibitor concentration and non closing behavior shown by loops indicates that layer has a porous structure [Duan et al., (2008)]. This capacitive loop indicates that the corrosion of mild steel is controlled by a charge transfer process. The presence of MRE increases the charge transfer resistance (Rct). This is mainly due to formation of protective film on the electrode surface results in decreasing the extent of dissolution reaction [Singh, (2008)]. The charge-transfer resistances

(Rct) were calculated by the difference in impedance at the lower and higher frequencies by using following formula [I.Ahmed, et al., (2010)].

IE% = (Rct (inh) – Rct/ Rct (inh)) × 100

Where Rct and Rct (inh) are charge transfer resistance in absence and presence of inhibitor respectively. With increase in inhibitor concentration Cdl decreases and the decreased value of Cdl is due to the adsorption of the MRE, which forms protective layer on metal surface (Abdel – Gaber et al., 2009). As the thickness of the protective layer increases, the inhibition efficiency also increases, since more inhibitor molecules will electrostatically adsorb on the electrode surface, resulting in a noticeable decrease in Cdl [Loto, (2011)]]. Double layer capacitance can be calculated by following relation

Cdl = 1 / 2π fmaxRct

Where fmax is the frequency at which the imaginary component of the impedance is maximal and Rct is charge transfer resistance. The inhibition efficiency calculated from EIS showed the same trend as those obtained from galvanostatic polarization measurement.

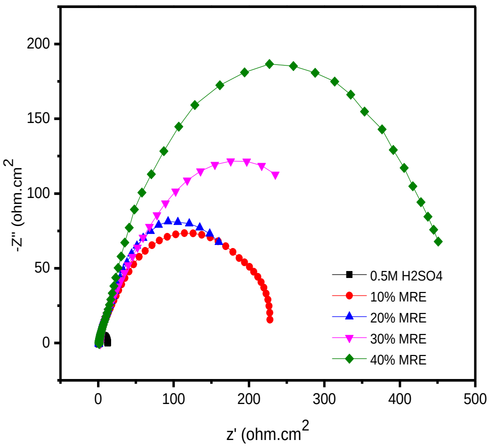


Figure 2a : Nyquist plots for mild steel in 0.5 M H2SO4 and in the presence of various concentrations of MRE at 298 K

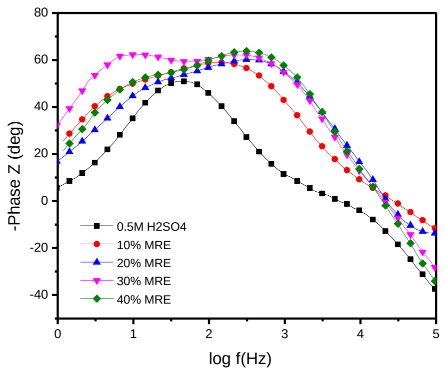


Figure 2b: Bode plot for mild steel in 0.5 M H2SO4 and in presence of MRE at 298 K

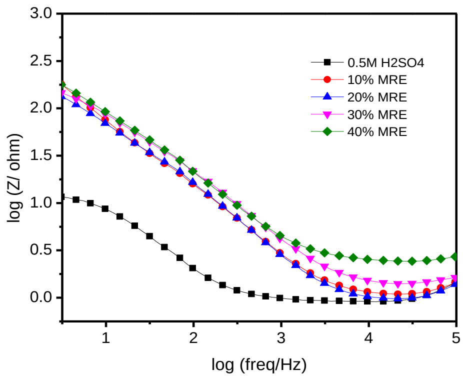


Figure 2c: bode plot for mild steel in 0.5 M H_2SO_4 and in presence of MRE at 298 K

Table. 2: EIS data for mild steel in 0.5 M H_2SO_4 in presence and absence of different concentrations of inhibitor MRE(Morus rubra Extract).

Solution	**conc.(v/v)%**	**R_{ct}**	**C_{dl}**	**F_{max}**	**%IE**
H_2SO_4	**0.5M**	**26.064**		**9.034**	**-**
MRE	**40%**	**497.29**	**1.742**	**183.8**	**95**
	30%	**384.01**	**3.411**	**121.5**	**93**
	20%	**261.21**	**7.616**	**80.03**	**90**
	10%	**249.12**	**8.022**	**71.7**	**89.4**

The results from EIS parameters shown in Table 3 reveals that the charge transfer resistance (Rct) and double layer capacitance (Cdl) values decrease with increasing inhibitor concentration this leads to an increase of % IEEIS.

3.4. Effect of Temperature

The effect of temperature at 298 K to 328 k has been studied on MS in 0.5 M H2SO4 the absence and presence of concentration the inhibition efficiency decreases and corrosion rate increases with increasing the temperature in both the condition i.e. with and without inhibitor [Singh et al., (2016)]. The dependence of corrosion rate on temperature can be represented by Arrhenius equation:

Icorr = k exp (- Ea/RT)

Where, Ea is the activation energy, T is the absolute temperature, k is the Arrhenius pre- exponential factor and R is universal gas constant. A plot of natural logarithm of the corrosion current density versus inverse of temperature (1/T) (fig. 3) gave a straight line with slope and intercept. The value of Ea can be calculated from the slope and shown in table 3. The value of Ea is higher in presence of MRE inhibitor then of uninhibited solution. With increase in concentration of inhibitor higher value of activation energy is attributed to its physical adsorption. Increase in activation energy causes decrease in the adsorption on the mild steel with increase in temperature [Orubite and oroka, (2008)]. With decrease in adsorption, desorption of inhibitor molecule starts, due to which metal surface came in contact with environment which leads to increase in corrosion rate with increase in temperature.

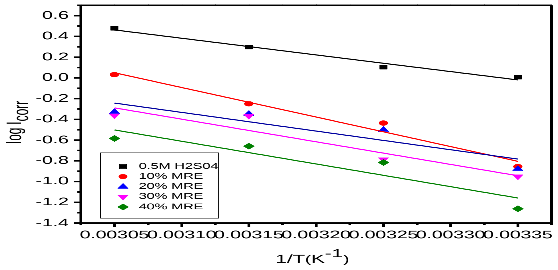


Figure. 3: Arrhenius plot of variation of log Icorr vs. 1/T

3.5 Adsorption Isotherm

Adsorption isotherms gives idea about the interaction of the adsorbed molecules with the electrode surface (Hussin and Kassim, 2011) Through adsorption isotherm adsorption of inhibitor on metal surface can be interptrated. Mode of adsorption of inhibitor. To study the mode of adsorption of MRE on the surface of mild steel, different adsorption isotherms were tested for the best fit. In the present study, the best fit was obtained with Langmuir isotherm which is given by

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

Where, C is concentration of the adsorbate, θ is the fraction of surface coverage and Kads is equilibrium constant for the adsorption process and depends on the temperature and extent of adsorption. The value of correlation constant (R2) obtained in the plots of C/θ versus C equal to or close to 1 indicates that Langmuir Adsorption Isotherm is followed for a particular adsorption process which is shown in Fig. 4. It indicates that MRE get adsorbed on typical adsorption site at the metal/solution interface [Ibrahim, et al., (2012)]. From the intercept values obtained from this graph, the value of adsorption equilibrium constant, Kads was calculated at the four temperatures as per equation given above.

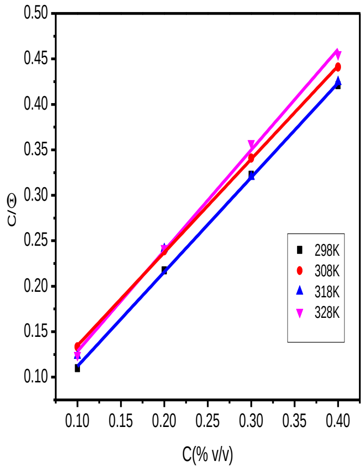


Figure. 4: Langmuir adsorption isotherm for mild steel in 1M H2SO4 containing different concentrations of MRE at 298-328.

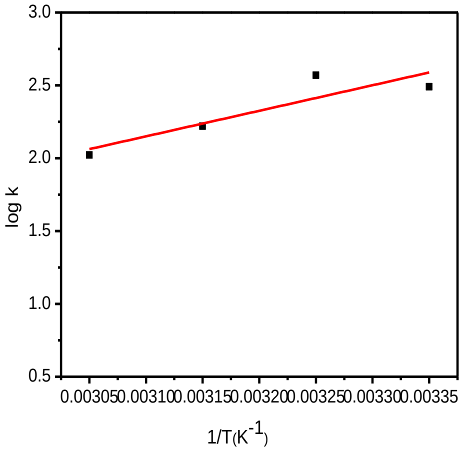


Figure 5: Plot of log K vs 1/T for the adsorption of MRE

Table.3: Adsorption parameters at different temperatures studied for MRE

Temperature	**-Δ H (kJ mol^{-1})**	***log K$_{ads}$* (L mol^{-1})**	***-Δ G$_{ads}$* (kJ mol^{-1})**	***-Δ S$_{ads}$* (J K^{-1} mol^{-1})**
298k	**-34.65**	**2.265**	**22.87**	**42.147**
308k		**2.065**	**22.10**	**53.37**
318k		**1.753**	**20.62**	**43.32**
328k		**1.491**	**20.31**	**40.73**

Different thermodynamic parameters are calculated after finding the best fit for the degree of surface coverage . These thermodynamic parameters are Gibbs Free Energy of Adsorption, ΔG°_{ads}, Enthalpy of Adsorption, ΔH°_{ads}, and Entropy of Adsorption, ΔS°_{ads}. The kinetic parameter include the calculation of Energy of Activation, E_a, which explains the effect of temperature on the adsorption process. All these parameters are related to the equilibrium adsorption constant, K_{ads}, which is derived from the equation of the adsorption isotherm given above.

The free energy of adsorption (ΔG°_{ads}) can be calculated using following equation

$$(\Delta G^{\circ}_{ads}) = -2.303\, RT\, log(55.5\, K_{ads})$$

Where 55.5 M is the morality of water under standard conditions, R is the gas constant and T is the absolute temperature. In the present study, negative value of ΔG°_{ads}(Table 3) clearly indicates the spontaneous adsorption of MRE on mild steel surface [Muthukrishnan et al., (2013)]. Heat of adsorption and entropy of adsorption are important parameters to understand adsorption mechanism of inhibitor on metal surface. ΔH°_{ads} can be correlated to equilibrium constant by the very popular Van't Hoff Equation

$$\frac{dl_nK_{ads}}{dT} = \frac{\Delta H^{\circ}_{ads}}{RT^2}$$

When log Kads is plotted against $1/T$, we get a straight line with slope equal to $\frac{\Delta H^{\circ}_{ads}}{2.303R}$ and intercept gives the value of integration constant. Thus, enthalpy of the adsorption process can be calculated from slope of this graph shown in Fig.5 .The values of ΔH°_{ads} and ΔG°_{ads} so obtained from above equations , respectively can now be substituted in equation given below to calculate the entropy of the adsorption process by using equation

$$\Delta G^{\circ}_{ads=} \Delta H^{\circ}_{ads} - T\Delta S^{\circ}_{ads}$$

It can be seen from the thermodynamic parameters that MRE is able to inhibit the corrosion of mild steel by getting physiosorbed on the surface of mild steel and thereby blocking the process of corrosion. The values of adsorption equilibrium constant Kads, decrease when the temperature is increased representing that the adsorption of inhibitor decrease at higher temperature, thereby providing less shielding and hence less protection from the aggressive action of the acid. Additionally, when the standard free energy of the reaction is of the order of 20 kJ mol–1, then it is said that the inhibitor molecules are "physiosorbed" [Benali et al., (2007)]. As can be seen from Table.3, it can be deduced that MRE is being physiosorbed on the surface of mild steel to provide protection against corrosion. A plot of ln Kads versus 1/T gives a straight line as shown in figure.5. From this slope of straight line, the change in Enthalpy of Adsorption (ΔH°_{ads}) is calculated which is depicted in table 3. The negative value of enthalpy behavior can be interpreted on the basis that at high temperature more desorption of adsorbed inhibitor occurred from mild steel surface [Zielinski, (1981)]. The value of Δ Sads are negative, because exothermic adsorption process is to be associated with a decrease in entropy orderliness increases as inhibitor molecules adsorb on metal surface, resulting in a decrease in entropy.

3.5 Surface characterization

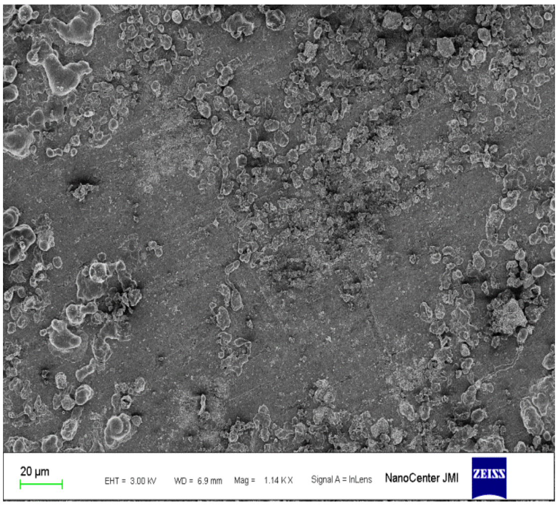
From SEM and AFM images surface morphology of mild steel in absence and presence of MRE can be seen clearly in Fig. 6(a–c). Fig. 6(a) shows the MS sample immersed in acid solution without inhibitor. In this image the MS surface is seems to be have some cracks and pits due to acid attack. The MS immersed in acid solution containing inhibitor are shown in Fig. 6(b, c). The inhibitor treated MS surfaces are smooth and protected from acid attack.

In addition to the surface study, through AFM analysis average surface roughness of MS in the absence and presence of MRE in 0.5 M H2SO4 was calculated. It is also a powerful technique to study the influence of inhibitor on metal surface. In the absence of MRE, the surface displayed an extremely rough topography (Fig. 7(a)) and the average surface roughness is 964 nm

Table. 4: Roughness data from AFM measurement for MS surface in 0.5M H2SO4 without and with 40% and 10% inhibitor:

Compound	Conc.	Roughness area(metal)nm
Plain MS	-	125.5
H2SO4	0.5M	964.4
MRE	40%	228.7
MRE	10%	327.2

In the presence of MRLE the MS shows smoother surface (Fig. 7(b, d)) and the average surface roughness value obtained at 10% and 40% concentration of inhibitor are 327.2 nm, 228.7 nm, respectively. Both the surface analysis techniques suggest that MRE act as efficient inhibitor



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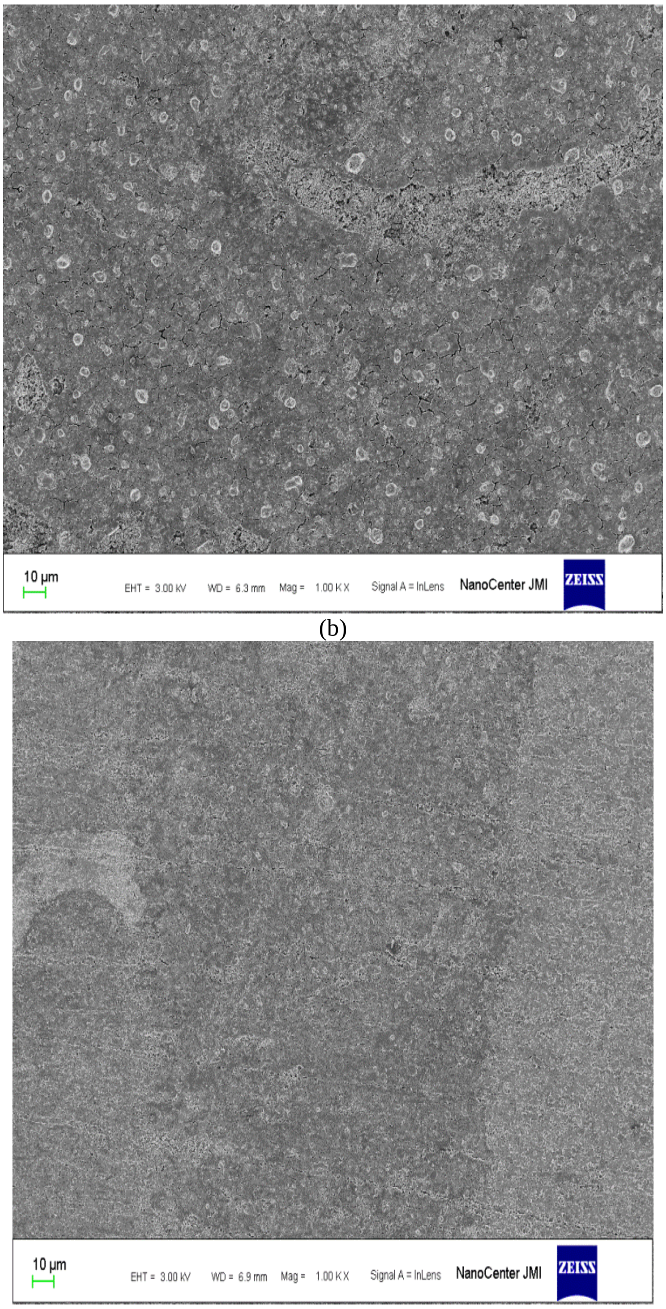
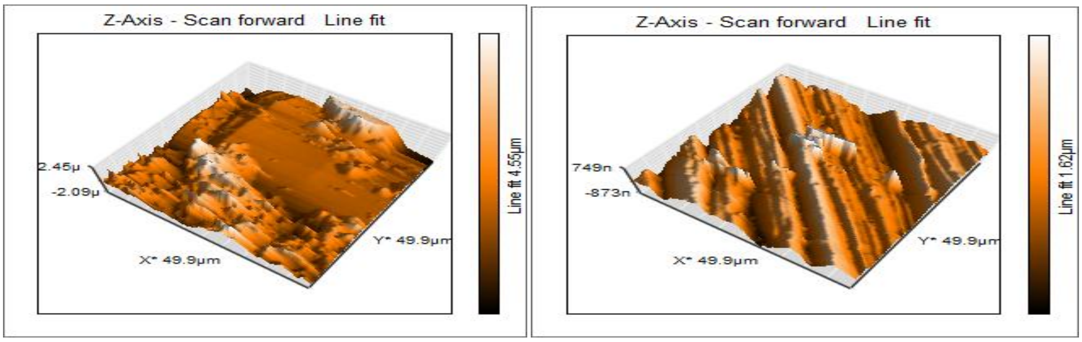


(b)

(c)

Figure.6: SEM images of mild steel (a) in 0.5 M H_2SO_4 (b) In 10% MRE (c) 40% MRE.



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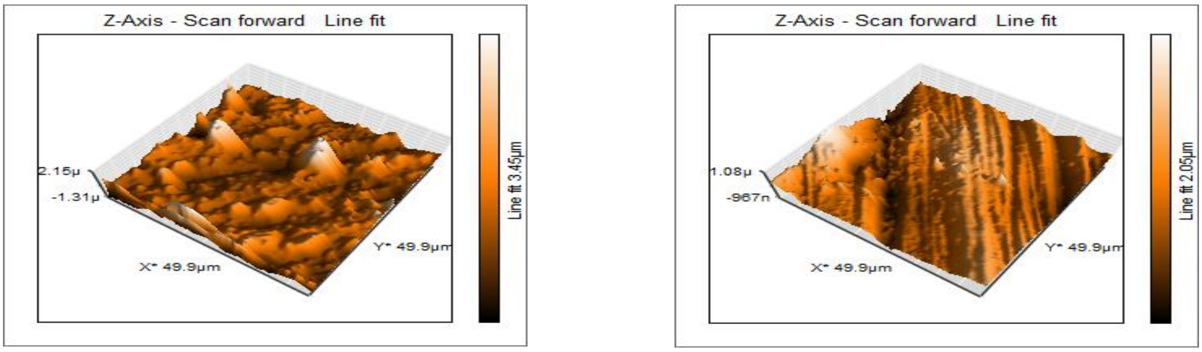

(b)

Figure 7: (a) 10% MRE (b) 40% MRE

4. Conclusion

Morus rubra leaf extract was found to be an effective inhibitor for mils steel corrosion in H2SO4. With increase in concentration of Morus rubra leaves extract inhibition efficiency of the extract and with increase in temperature inhibition efficiency decreases. At the highest extract concentration, inhibition efficiency reaches to 94.6%. from polarization curve measurement it was analyse that morus rubra leaves extract was a mixed type of inhibitor. From EIS plot it was concluded that the charge transfer resistance increases with increase in concentration of extract. The adsorption model followed the Langmuir isotherm .

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