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Study of Mild Steel Corrosion in Sulphuric acid medium by *Moringa oleifera* leaf extract by Electrochemical and Surface Analysis Studies

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Abstract : The inhibitive effect of *Moringa oleifera* leaf extract on mild steel corrosion in 1N H_2SO_4 solution was studied using electro chemical techniques and gravimetric method. The weight loss results showed that the plant extract was an excellent corrosion inhibitor. The inhibition efficiency increased with increase in concentration of the plant extract. The highest inhibition efficiency of 96% was observed with 8ml of plant extract in acidic medium. Immersion period was carried out to optimize the period of immersion. The surface studies such as FT-IR, scanning electron microscopy (SEM), EDAX were carried out to confirm the protective layer formed on the metal surface. The inhibition effect is due to the adsorption of active molecules leading to the formation of a protective layer on the surface of mild steel.
Keywords : Acid corrosion inhibition, *Moringa oleifera*, Mild steel, Weight loss, Polarization, Impedance spectra.

1. Introduction

"Corrosion of metals such as mild steel is an electrochemical reaction which can cause the degradation and batter of the physical and chemical properties of the metal"[1]. The use of inhibitor is found to be one of the most practical methods to protect metals from corrosion, especially in acid solutions [2]. Most of the inhibitors having hetero atoms such as O, N, S and multiple bonds in their structure. They are adsorbed on the surface of metal [3-4]and also act as a barrier between metal surface and electrolytic solution. Even though the resistivity of corrosion is good and most of the compounds are well toxic[5]. Hence the researchers are focusing on the "Green corrosion inhibitors " or Eco friendly inhibitors which shows good inhibition efficiency with low risk of environmental pollutions[6]. Until now several extract of plants such as Nypa fruticans Wurmb, Datura metel, Ricinus communis, Occimum viridis, Musa sapientum, Phyllanthus amarus, Fenugreek, Vernonia amygdalina, Justicia gendarussa, Telferia occidentalis, Azadirachta indica and Hibiscus sabdariffa as well as extracts from the seeds of Garcinia kola, Murraya koenigii, fruit peel, Uncaria gambir, Punica granatum, Piper guineense, Punica granatum, Tageteserecta[7-23] have been investigated for corrosion inhibition of metals in acidic medium. The inhibitors might be adsorbed on the surface of the metal by the following ways such as electrostatic interaction, donor-acceptor interaction, interaction between unshared pair of electrons of heteroatoms and vacant d orbital of surface metal atoms [24]. The trend of an inhibition efficiency increased with increase in

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concentration of the plant extract [25]. In the present study, the corrosion inhibiting effect of ethanol extract of Moringa oleifera as a green inhibitor on the mild steel in 1N sulphuric acid solution was investigated. The corrosion performance was studied using different techniques like Potentiodynamic polarization, electrochemical impedance spectroscopy and weight loss methods. The surface studies such as FT-IR, scanning electron microscopy, EDAX were carried out to confirm the protective layer formed on the metal surface.

2. Experimental

2.1 Experimental of specimens

Mild steel specimens having weight compositions of carbon-0.116%, Sulphur-0.007%, Phosphorus-0.006%, Manganese-0.85%, Silicon-0.05% of the dimensions 4cm×0.4cm×0.2cm were polished to mirror finish, degreased with acetone, dried and then used for weight loss method. In addition some coupons were cut into $1cm^2$ which were used for electrochemical and surface examination studies [26].

2.2 Preparation of extract

Leaves of Moringa oleifera were air dried at room temperature and powdered. The 20% of ethanolic extract could be prepared using magnetic stirrer and concentrated at 60C, the extract was filtered. The filtrate was then used for the study[27].

2.3 weight loss method

The polished and weighed samples of mild steel coupons in triplicate were dipped in 100ml experimental solution with and without different concentration of extracts for about 1hr, 2hrs, 4hrs and 24hrs. The test was carried out at room temperature. Then the specimens were taken out, washed with distilled water, dried and then weighed [28]. Inhibition efficiency of Moringa oleifera extract for the corrosion of mild steel was calculated according to the following equation

$$\text{Inhibition Efficiency (\%)} = \frac{W_B - W_I}{W_B} \times 100$$

Where, W_B and W_I are weight loss per unit time in the absence and presence of inhibitors.

2.4 Electrochemical Polarization study

Measurements were carried out using an (Bio-logic SP300 with EC lab software) a three electrode assembly. The working electrode was used as rectangular specimens of mild steel with one face of the electrode of constant $1cm^2$ area exposed. Platinum as counter and saturated calomel as reference electrode were used [29-30]. Polarization curves were recorded at 1mv sec-1. The Potentiodynamic current potential of 0.025 mV. The results such as corrosion potential (E_{corr}) and corrosion current (I_{corr}) were determined from E vs log I plots.

2.5 Electrochemical impedance spectroscopy

Electrochemical impedance spectroscopy was carried out by using a Bio-logic SP 300 with EC lab software. The cell setup was the same as that used for polarization measurements. Impedance measurements were carried out in a frequency range of 100000Hz to 0.010Hz using amplitude of 10mV peak using AC signal at the open circuit potential. The charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) values were calculated.

2.6 Surface characterization studies

The mild steel specimens were immersed in the acid solutions with and without inhibitor. The nature of the film formed on the surface of the specimens was analyzed by various surface analysis techniques.

2.6.1 FT-IR spectroscopy

Fourier transform infrared spectra were recorded using JASCO FT-IR 6300 model spectrophotometer. The film formed on the metal surface with and without inhibitorin 1N sulphuric acid solution were studied.

2.6.2 SEM and EDAX

The test specimens of the size 11cm^2 were exposed in 1NH_2SO_4 solution in the absence and presence of plant extract for a period of 1hour at room temperature. The surface of the corroded metal and inhibited metal were analyzed using FEI quanta FEG 200-high resolution field emission electron microscope with energy dispersive X-ray analyzer for chemical analysis [31]. These studies further supported the gravimetric and electrochemical studies, which confirmed the formation of protective layer over metal surface.

3. Result and Discussion

3.1 Mass-loss measurements

Corrosion parameters such as corrosion rate and inhibition efficiency were studied for four different concentrations of inhibitor ranging from 2ml to 8ml in 1N H_2SO_4 for the time intervals 1hr, 2hrs, 4hrs and 24hrs tabulated in table 1 & 2. It was shown that with rice in concentration of Moringa oleifera leaf extract from 2ml to 8ml, the weight loss of mild steel decreased while the inhibition efficiency increased from 70% to 96% in 1N H_2SO_4 medium for about 1hr Moringa oleifera has the highest inhibition efficiency of about 96% at the optimum concentration of 8ml. The obtained results indicate that the Moringa oleifera leaf extract could act as an excellent corrosion inhibitor. Even with increase in immersion period, Moringa oleifera leaf extract showed maximum inhibition efficiency. This could be due to the maximum adsorption of inhibitor molecules on to the metal surface.

Table 1: Corrosion rates (CR) for mild steel immersed in various acid solutions in the absence and presence of inhibitors and the inhibition efficiencies (IE) obtained by weight loss method.

Medium	Volume of Extract (ml)	Immersion period			
		1 hr		24 hrs	
		CR (mm/y)	IE (%)	CR (mm/y)	IE (%)
1N H_2SO_4	Blank	112.56	-	64.65	-
	2	13.15	88	44.74	30.7
	4	12.59	89	29.24	54.7
	6	6.24	94	17.40	73
	8	4.01	96.4	2.54	96

Table 2:Effect of immersion period on the inhibition efficiency of best inhibitor system of 8 ml extract

Medium	Volume of extract (ml)	Corrosion Parameters	Immersion period			
			1 hr	2 hrs	4 hrs	24 hrs
1N H_2SO_4	Blank	CR(mm/y)	112.56	87.09	95.26	64.65
		IE (%)	-	-	-	-
	8 ml	CR(mm/y)	4.01	3.67	1.78	2.54
		IE (%)	96.4	95.7	98.1	96.0

Table 3: The electrochemical polarization parameters for mild steel in 1N H_2SO_4 in the absence and presence of inhibitor at different concentrations.

S.No	Concentration (ml)	$-E_{corr}$ (mV)	I_{corr} (μA)	βa (mV)	βc (mV)	CR (mm/y)	IE (%)
1	Blank	487	1438	40	26	16.85	-
2	2	488	1022	22	34	11.98	28.92
3	4	489	911	33	44	10.68	36.62
4	6	486	785	14	23	9.21	45.34
5	8	476	624	13	17	7.32	56.57

3.2 Analysis of electrochemical Potentiodynamic polarization

Polarization study has been used to study the protective film formed on the metal surface. The Potentiodynamic polarization curves of mild steel immersed in 1N H_2SO_4 acid environment in the absence and presence of inhibitor are shown in figure 1. The corrosion parameters namely corrosion potential (Ecorr), corrosion current density (Icorr), Tafel slopes (ba&bc), corrosion rate (CR) and inhibition efficiency (IE%) are given in table 3.Ecorr values shifted to positive potential with increase in concentrations of Moringa oleifera leaf extract [32]. The corrosion current densities reduced from 1438µA to 624µA with increase in concentration of the inhibitor. Although, there is no notable difference in the Ecorr range attained in the absence and presence of inhibitor. The plots shows linear shape at lower current densities due to protective layer formed on the metal surface and decreased the transfer of electrons at the interface. The protection can be attributed due to the nitrogen atom which donates the loan pair of electrons to surface of the metal for easy adsorption and hence reduces the corrosion rate [33]. As it is seen from table 3 the maximum inhibition efficiency was found to be 56.57% in 1N H_2SO_4 at 8ml concentration of inhibitor at room temperature. Inhibition efficiency increased with increasing inhibitor concentration and also decreased rate of corrosion.

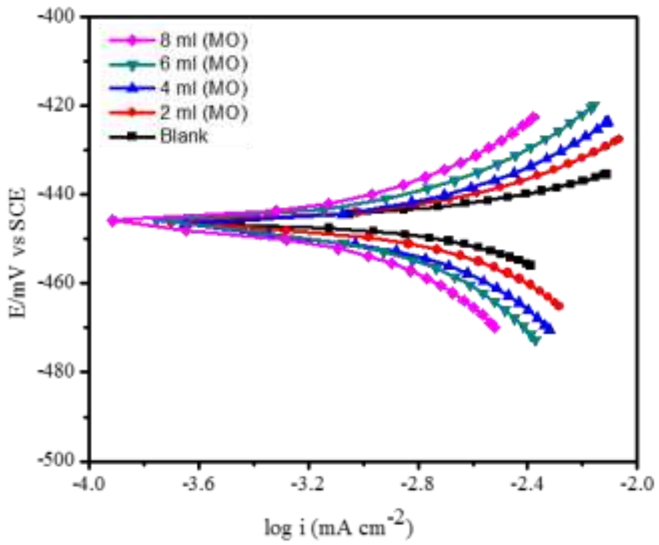

Figure 1.Tafel plots for mild steel in 1 N H_2SO_4 in absence and presence of different concentration of inhibitor

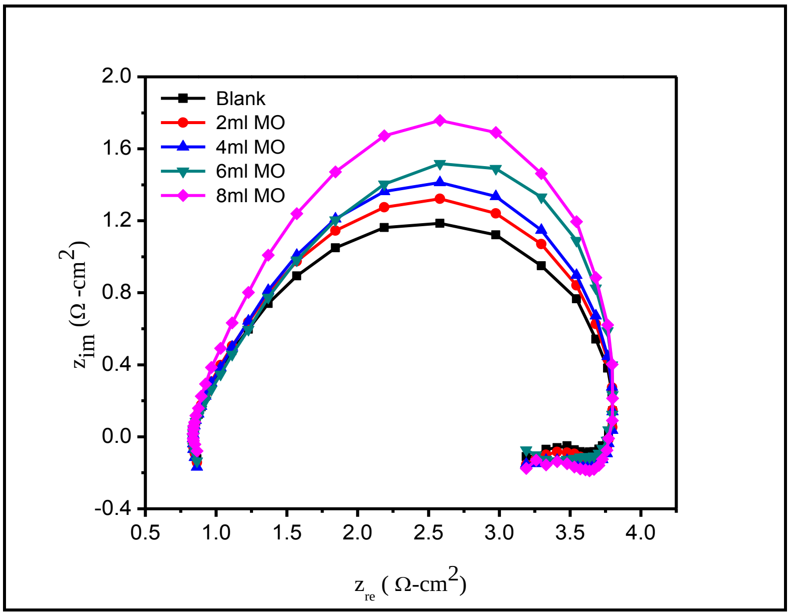

Figure 2.Nyquist plots for mild steel in 1 N H_2SO_4 in absence and presence of different concentration of inhibitor

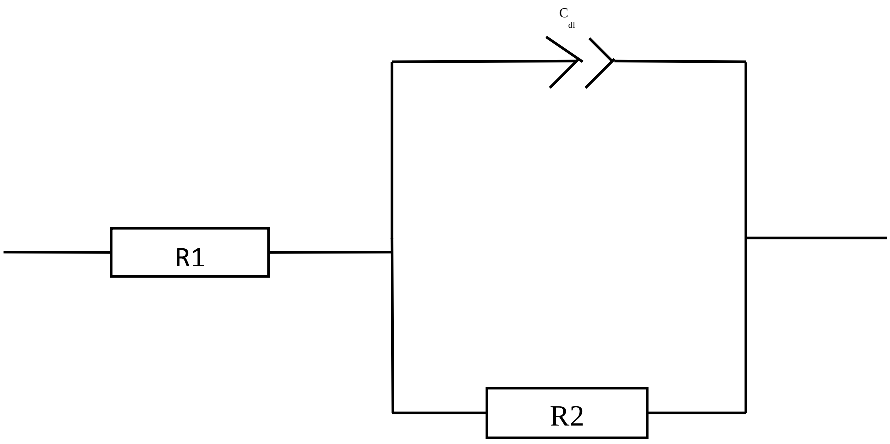

Fig 3 Equivalent circuit for fit the impedance spectra

3.3 Analysis of electrochemical impedance spectroscopy

Electrochemical impedance measurements for mild steel corrosion in1N H_2SO_4 in the absence and presence of various concentrations of Moringa oleifera leaf extract at room temperature is represented in Fig 2. The semicircle impedance diagram suggested that mild steel corrosion in 1N H_2SO_4 is mainly controlled by charge transfer process. It is observed that, the size of the semicircle increased with increase in inhibitor concentrations. The impedance spectra were investigated by fitting to the electrochemical equivalent circuit (fig 3), which comprises a solution resistance (R_S), double layer capacitance (C_{dl}) in parallel with charge transfer resistance (Rct). The values of charge transfer resistance calculated from difference between the lower and higher frequencies of impedance. The values of inhibition efficiency

(IE%) was calculated by following equation,

$$IE(\%) = \left(\frac{R_{ct(inh)} - R_{ct}}{R_{ct(inh)}}\right) \times 100$$

Where $Rct_{(inh)}$ and Rct are charge transfer resistances in 1N H_2SO_4 medium with and without inhibitors respectively.

Table 4: The electrochemical impedance parameters for mild steel in 1N H_2SO_4 in the absence and presence of inhibitor at different concentrations.

Concentration (ml)	Rct (ohm.cm^2)	C_{dl} (µF/cm^2)	IE (%)
Blank	1.79	31.05	-
2	2.32	26.50	29.9
4	2.82	19.91	36.5
6	2.97	18.35	39.8
8	4.97	6.23	64.0

The impedance parameters namely charge transfer resistance (Rct), double layer capacitance (C_{dl}) and inhibition efficiency (IE%) derived from Nyquist plots are given in table 4. From, the table it is clear that with increase in Moringa oleifera leaf extract concentration, the Rct values increases and the C_{dl}values decrease suggest the inhibitor molecules adsorbed on the metal surface. Rct increases from 1.79 to 4.97 Ω cm2 and C_{dl} decreases from 31.05 to 6.23 µF/cm2with increase in concentration of Moringa oleifera leaf extract. The increase in charge transfer resistance resulted, increase in inhibition efficiency. The Rct is inversely proportional to the corrosion rate. The C_{dl} values were obtained at the frequency $f$max at the imaginary component of impedance by following equation

$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}}$$

As a result inhibition efficiency for Moringa oleifera leaf extract has increased from 29.9 to 64%. The highest 64% inhibition efficiency was observed at 8ml of the plant extract These values was in good agreement with those obtained from both Potentiodynamic polarization and weight loss measurements.

3.4 FTIR spectroscopy

Infrared spectroscopy studies were carried out to qualitatively evaluate the main functional groups responsible for binding with metal surface. This method provides information about the functional groups that may be present in the structure of the adsorbents. FT-IR of Moringa oleifera leaf extract exhibited peaks at 3471, 2919, 2854 and 1024 cm^{-1}(Fig.4a). FT-IR spectra of film formed on the surface metal exhibited prominent peaks at 3514, 2914, 2864 and 1035 cm^{-1}(Fig. 4b). It is seen from the spectrum that theO-H stretching vibration present in Moringa oleifera leaf extract has shifted from 3471cm^{-1} to 3514 $cm^{-1.}$ Also N-H stretching in MO extract of frequency 2919cm^{-1} has shifted to 2941cm^{-1}.The peak at 2854cm^{-1} was shifted to 2864cm^{-1}correspond to C–H (methoxy compounds) stretching vibration respectively. The band shifted from 1024 cm^{-1} to 1035 cm^{-1} developed for C–C and C–N stretching [34-37]. This indicates Fe^{2+} has coordinated to the lone pair of oxygen and nitrogen atoms resulting in the formation of a complex on metal surface.

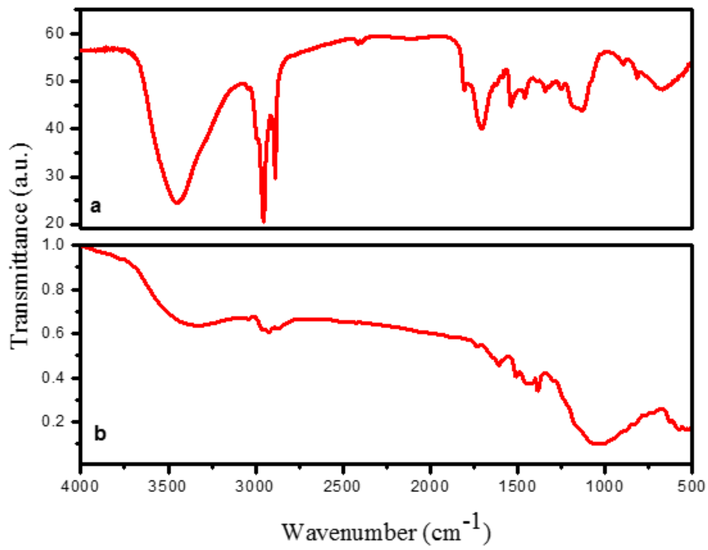

Fig 4 FTIR spectra for (a) Moringa oleifera extract and (b)metal after immersion in 1 N H_2SO_4 + 8ml extract.

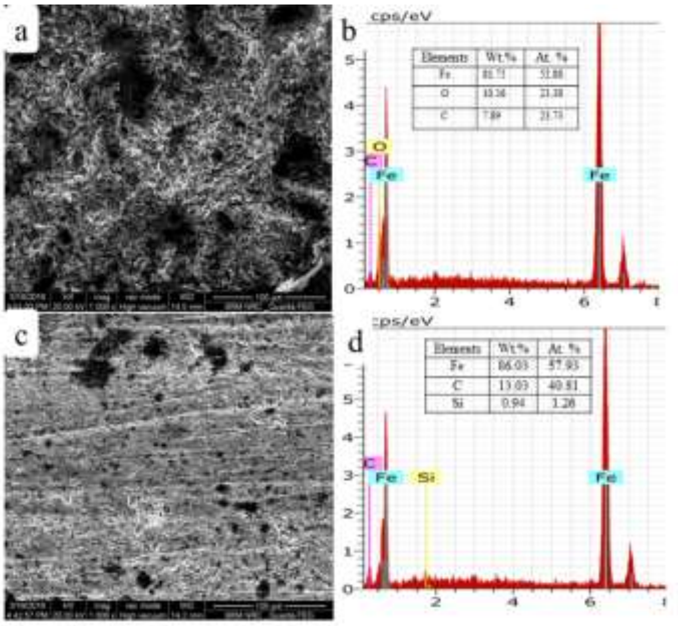

Figure 5: SEM and EDAX images of mild steel surface after immersion in1N H_2SO_4 (a & b) absence and (c & d) Presence of inhibitor

3.5 SEM and EDAX

Surface examination by SEM and EDAX Fig. 5(a and b) performed for C-steel surface immersion in 1N H2SO4, for a period of 1 h, in the absence and presence of Moringa oleifera leaf extract respectively. The SEM micrographs (Fig.5a) shows that mild steel coupon was highly damaged and its surface is full of roughness and cavities in the absence of inhibitor solution. The corresponding EDX spectra (Fig.5b) observed (Fe, C and O) Fe and C peaks tells metal constituents, O peak shows presence of corrosion products such as iron oxides or hydroxides. However, the tiny scratches happening are seeable on the surface of the metal in the presence of the inhibitor (Fig. 5c). The EDAX spectra (Fig.5d) shows Fe, C and Si confirmed that Fe is only the major element present in the study. No oxygen peak was detected this indicates that corrosion products are restricted due to inhibitor layer adsorbed on the metal surface. Additional weak peak of Si being from alloy constituents. The reduction in the amount of corrosion products indicates the anti-corrosion effect of these Moringa oleifera leaf extract for mild steel in 1N H_2SO_4[38-41].

4. Conclusions

The influence of Moringa oleifera plant extract on the corrosion of mild steel was investigated in Sulphuric acid medium. From the results obtained it could be concluded as Moringa oleifera leaf extract showed increase in inhibition efficiency with increase in inhibitor concentration. It showed maximum inhibition efficiency of 96% for 8 ml of the extract. The inhibition efficiency does not change with increase in immersion period and showed maximum inhibition efficiency even for 1 day (24 hrs). The polarization study reveals that, Moringa oleifera lead extract act as a mixed type inhibitor. Surface studies confirmed protective film formed on the metal surface.

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