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The inhibition of mild steel Corrosion in 1N HCl solution by Melia Azedarach leaves extract

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ABSTRACT

The inhibition of the corrosion of mild steel in hydrochloric acid solution by the leaves Extract of Melia Azedarach (MA)has been studied using weight loss, electrochemical impedance spectroscopy and Potentiodynamic polarization techniques. Inhibition was found to increase with increasing concentration of the extract. Results indicate that MeliaAzedarach leaves Extract was an efficient natural corrosion inhibitor in the acidic solution. Polarization measurements showed that the studied inhibitor acts as mixed inhibitor in 1N HCl acid with significant reduction of cathodic and anodic current densities. The electrochemical impedance study further confirmed the formation of an adsorbed film on the mild steel. The SEM morphology of the absorbed protective film on the mild steel surface has confirmed the high performance of inhibitive effect of the plant extract.

Keywords: mild steel, EIS, SEM, Polarisation.

INTRODUCTION

The study of mild steel corrosion is theoretical and practical importance [1]. Mild steel is commonly used in industries because of cost-effective and easy fabrication, but it is prone to undergo corrosion in aggressive environmental conditions. Usually, sulphuric acid and hydrochloric acid are used in acid pickling, chemical cleaning to eliminate detrimental rust and scale in several industries. One of the significant methods of dealing with this problem is the usage of corrosion inhibitor in the aggressive media [2]. Severe corrosion attack is known to occur on metals in such aggressive environments. Aqueous solutions of acids are among the most corrosive media. The inhibition of corrosion on iron base alloys by organic additives has been studied widely [3-6]. A considerable work has been done on the study of organic compounds as corrosion inhibitors [7-8]. Most acid corrosion inhibitors are N, O, and S containing organic compounds [10-11]. But, unfortunately most of them are highly toxic to both human being and environment. Most of the synthetic organic compounds showed good anticorrosive activity, which are highly toxic to cause severe hazards to both human being and the environment during its application [12]. Hence the use of natural products which are eco-friendly and are being used as corrosion inhibitors has become need of the time. Now the development of corrosion inhibitor of natural source and nontoxic type has been considered more important and desirable [13-22]. Plant extracts contain mixtures of proteins, polysaccharides, polycarboxylic acids, tannin, alkaloids, and terpinoids and so forth [23]. Some investigators studied the plant extract and the derived organic species become more important as an environmentally benign, easily available and acceptable source for a wide range of inhibitor [24-25]. A large number of research studies have been devoted to the inhibitive performance of natural corrosion inhibitors on the corrosion of metals in various aggressive solutions showing that these extracts could act as potential corrosion inhibitors [26-42]. The present study aims at broadening the application of plant extracts for mild steel corrosion inhibition by investigating the inhibitive properties of MA extract for mild steel corrosion in 1N HCl using weight loss method, potentiodynamic polarization and electrochemical impedance spectroscopy technique.

MATERIALS AND METHODS

2.1 Preparation of Leaves Extract

The leaves of medicinal plants MA collects were taken and cut into small pieces, and they were shade dried in room temperature for few days and ground well into powder. From this, 25g of sample was refluxed in 200ml distilled water for 3 hour and kept overnight. The refluxed was then filtered carefully, the filtrate volume was made up to in 500ml using double distilled water which was the stock solution, and the concentration of the stock solution was expressed in terms of ppm.

From the stock solution, 05 - 25 ppm concentration of the extract was prepared using 1N hydrochloric acid. Similar kind of preparation has been reported in studies using aqueous plant extract in the recent years.

2.2 Preparation of Mild Steel Specimen

Mild steel strips containing the composition of (C-0.030%, Mn-0.169%, Si-0.015%, P-0.O31%, S-0.029%, Cr-0.029%, Ni-0.030%, Mb-0.016%, Cu-0.017%), and the reminder Fe, were mechanically cut into 4cm x 2cm x 0.1cm and were used for weight loss studies. The metal specimen were ground successively with (400, 600, 800, 1000, 1200 grade) grit silicon carbide paper (emery paper) followed to achieve mirror finish surface. For electrochemical studies, the mild steel strips of the same composition but with an exposed area of 1cm^2 were used, subsequently degreased with acetone and finally washed with deionized water, and stored in the desiccators. Accurate weight of the samples was taken using four digit electronic balance model (Shimadzu aay220).

METHODS

3.1 Weight Loss Methods

Mild steel specimens were immersed in 200ml of 1N HCl solution containing various concentration of the inhibitor in the absence and presence of mild steel for 24 hours. The weights of the specimens before and after immersion were determined using four digit model.

From the mass loss measurements, the corrosion rate was calculated using the following relationship.

$$\text{CR (mmpy)} = \frac{\text{K x Weight loss}}{\text{D x A x t (in hours)}} \qquad 1$$

Where, K = 8.76x10^4 (constant), D is density in gm/cm^3 (7.86), W is weight loss in grams and A is area in cm^2.

The inhibition efficiency (%) was calculated using equation (2) respectively,

$$\text{IE\%} = \frac{W_0 - W_i}{W_0} X100 \qquad 2$$

Where, W_0 and W_i are the weight loss in the absence and presence of the inhibitor.

3.2 Fourier Transform Infrared (FTIR) Spectrum

FTIR spectra were recorded with BRUKER ALPHA 8400S spectrometer. The film was carefully removed, mixed thoroughly with KBr made into pellets and FTIR spectra were recorded.

3.3 Potentiodynamic Polarization Method

Potentiodynamic polarization measurements were carried out using CHI608D electrochemical work station analyzer. The polarization measurements were used to evaluate the corrosion current, corrosion potential and Tafel slopes. The experiments were carried out in conventional three electrode cell assembly with working electrode as mild steel specimen of 1 cm^2. Platinum electrode was used as counter electrode and calomel electrode was used as reference electrode. A time interval of 10-15 minutes was given for each experiment to attain the study state open circuit potential (OCP). The polarization was carried out from cathodic potential to anodic potential at a sweep rate of 1 mV per second. From the polarization curves, Tafel slopes, corrosion potential and corrosion current were calculated. The inhibitor efficiency was calculated using the formula:

$$\text{IE\%} = \frac{I_{Corr} - I^*_{Corr}}{I_{Corr}} X100 \qquad 3$$

Where I_{corr} and I^*_{corr} are corrosion current in the absence and presence of inhibitors.

3.4 Electrochemical Impedance Method

The electrochemical AC-impedance measurements were also carried out using CHI6608E electrochemical workstation analyzer. Experiments were carried out in three cells assembly as that used for potentiodynamic polarization studies. A sine wave with amplitude 10 mV on the steady state open circuit potential. The real part (z') and the imaginary part (z'') were measured at various frequencies in the range of 100 kHz to 10 MHz. A plot of z' versus z'' was made. From the plot the charge transfer resistance (R_{ct}) was calculated and double layer capacitance (C_{dl}) was calculated using formula:

$$C_{dl} = 1/2\pi f_{max} Rct \qquad 4$$

Where R_{ct} is charge transfer resistance and C_{dl} is double layer capacitance. The experiments were carried out in the absence and presence of different concentration of inhibitor.

$$\mathrm{IE\%} = \frac{\mathrm{R_{ct}} - \mathrm{R_{ct}^{0}}}{\mathrm{R_{ct}}} \mathrm{X100} \qquad 5$$

Where R_{ct} and R^{0}_{ct} are the charge resistance values in the inhibited and uninhibited solution respectively.

3.5 Scanning Electron Microscopy

The mild steel specimen immersed in blank and in the inhibitor solution for a period of one day was removed, rinsed with double distilled water, dried and observed in a scanning electron microscope to examine the surface morphology. The surface morphology measurements of mild steel were examined using (JEOL) computer controlled scanning electron microscope.

3.6 Phytochemical Screening

Phytochemical screening was carried out on the freshly prepared aqueous *MeliaAzedarach*leaves extract according to the common Phytochemical method described earlier. The different chemical constituent tested included alkaloids, terpenoids, sterols, triterpenes, anthraquinones and flavonoids.

3.7. Effect of immersion time

The prepared mild steel coupons were immersed in 100 ml of the test solution without and with the MA extract of various concentrations for 1h, 3 h, 5 h, 7 h and 12 h at room temperature. The weight of the coupons before and after immersion was determined. Inhibition efficiency of the mild steel was calculated.

3.8. Effect of temperature

The polished and pre – weighed specimens were suspended in 100 ml of the test solution without and with the addition of various concentration of the MA extract for 1h in the temperature range of 303 – 323K using water thermostats. The specimens were removed from the test solution after 1 h and washed with distilled water, dried and weighed. The inhibition efficiency was calculated from the weight loss.

RESULTS AND DISCUSSION

4.1 FT-IR spectrum of MA

FT-IR spectrum was recorded for MA leaves extract in order to confirm the presence of various compounds which contributed in effective working of the inhibitor is shown in Figure. 1. The broad peak obtained at 3912 cm^{-1} can be assigned to N-H or O-H stretching. Another peak obtained at 3289 cm^{-1} may be strong C-H stretching vibration. Adsorption peak obtained at 2134cm^{-1} may be due to stretching of -C≡C- band. The peaks observed at 1651 cm^{-1} may be C=O bending vibration. Few peaks can also observed at 1411 cm^{-1}, 1374 cm^{-}1, 1073 cm^{-1}, which correspond to C=C and C-O stretching vibration. On the basis of the result it can be said that MA extract contain Nitrogen and Oxygen (N-H, C=N, C-N, O-H, C=O, C-O) in various functional group and aromatic ring , which make this extract attractive for being used as inhibitor.

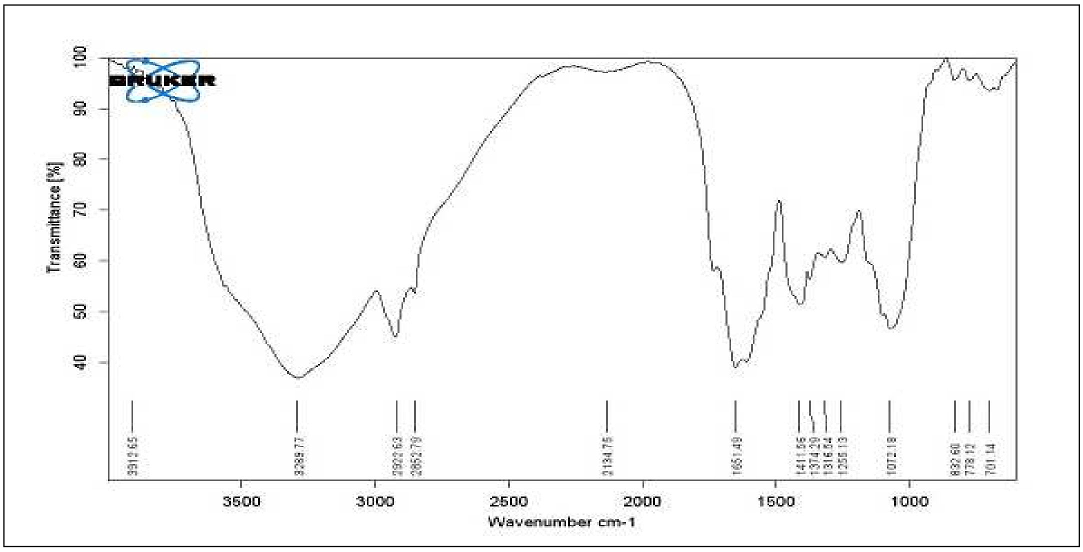

Figure. 1 FT-IR spectrum of Melia Azedarach leaves extract

4.2 Weight Loss Method

The weight loss method was done with concentrations of MA extract ranging from 5 to 25 ppm for mild steel in 1N HCl with various concentrations and the corresponding values of inhibition efficiency and corrosion rate are given in Table. 1. It was observed from the table that the corrosion rate was decreased and thus the inhibition efficiency was increased with increasing concentration of MA extract. The maximum inhibition efficiency of about 91.92% was achieved at 20 ppm of MA extract. This result indicated that MA extract could act as an excellent corrosion inhibitor.

Table. 1 Data from Weight Loss Method for MS corroding in 1N HCl solutions at various concentrations of MA extract

Conc. of MA Extract(ppm) Extract(ppm)	Corrosion Rate (mmpy)	Inhibition Efficiency (%)
Blank	0.0174	*
5	0.0016	63.33
10	0.0081	53.33
15	0.0058	66.67
20	0.0017	91.92
25	0.0034	80.01

4.3 Potentiodynamic polarization Studies

Potentiodynamic anodic and cathodic polarization scans were carried out in 1N HCl with different concentrations of MA extract. The potentiodynamic polarization curves for mild steel in 1N HCl with and without inhibitor (extract) are shown in Figure. 2. It is evident from the Figure that the anodic and cathodic curves for mild steel inhibited with extract were shifted towards positive potential region compared to the blank metal immersed in 1N HCl. The corrosion parameters such as corrosion potential (E_{corr}) and corrosion current density (I_{corr}), obtained from Tafel plots are given in Table. 2. From the Table. 2, it is observed that the I_{corr}values are found to decrease with increase in the inhibitor concentrations, ranging from 5 to 25 ppm.

Polarization study has been used to detect the formation of protective film on the metal surface. The shift in the values of corrosion potential (E_{corr}) for MA extract is not significant. This observation clearly showed that the inhibition of mild steel in the presence of the extract control both cathodic and anodic reactions and thus the inhibitor acts like mixed type inhibitors. The corrosion rates calculated for the mild steel specimens with different concentrations of the inhibitor are also given in Table. 2. As can be seen, the mild steel with 20 ppm concentration of the inhibitor showed excellent resistance than when compared to other concentrations of the inhibitor. For instance, the corrosion rate for mild steel without inhibitor (blank) was found to be 1.3913 mmpy and it was minimized by inhibitor with different concentrations like 5, 10, 15, 20 and 25 ppm to lower values of 0.9287, 0.6501, 0.4643, 0.1393 and 0.2786 mmpy, respectively. The inhibition efficiency values in the Table. 2 showed that the MA extract acted as very effective corrosion inhibitor for mild steel in HCl solution and its capacity of inhibition increased with increasing concentration.

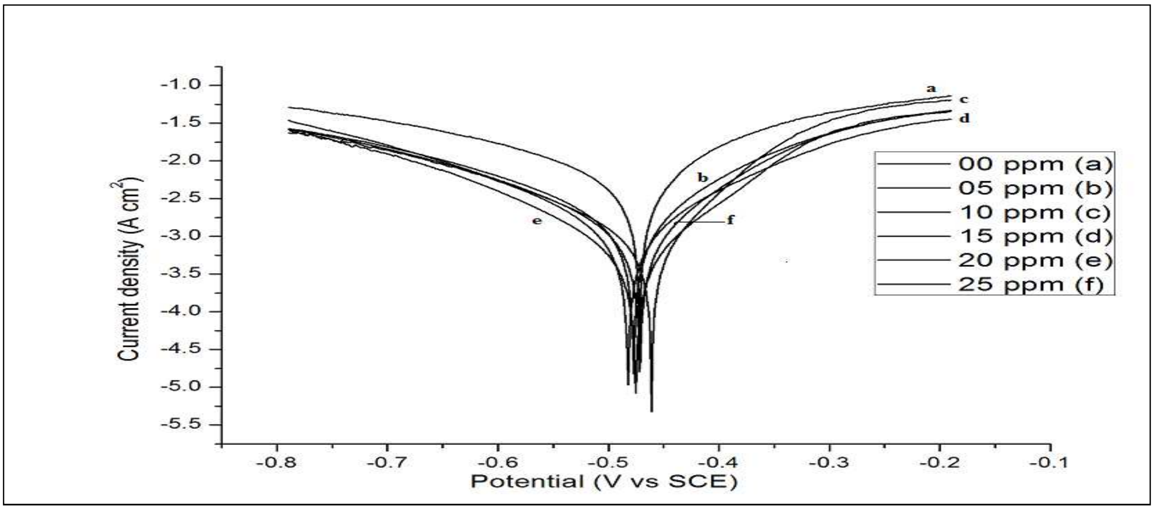

Figure. 2 Potentiodynamic polarization curves for mild steel in 1N HCl solution in the absence and presence of different concentrations of MA leaves extract

However, the maximum inhibition efficiency of 90.91% was achieved at 20ppm. From these findings, it is concluded that 20ppm is an optimum concentration of the inhibitor where the rate of corrosion is found to be maximum.

Table. 2 Potentiodynamic polarization parameter for mild steel in 1N HCl solution containing various concentrations of MA leaves extract

Conc. of MA (ppm)	E_{corr} (mV) vs.(SCE)	I_{corr}(mA cm^{-2})	CR (mmpy)	b_c (mV/dec)	b_a (mV/dec)	IE(%)
0	-472	6.486	1.3913	208	168	*
5	-477	1.642	0.9287	176	129	74.68
10	-461	0.899	0.6501	166	86	86.13
15	-482	1.235	0.4643	160	138	80.95
20	-475	0.589	0.1393	143	94	90.91
25	-472	1.145	0.2786	168	111	82.34

4.4 Electrochemical Impedance spectroscopy (EIS) Studies

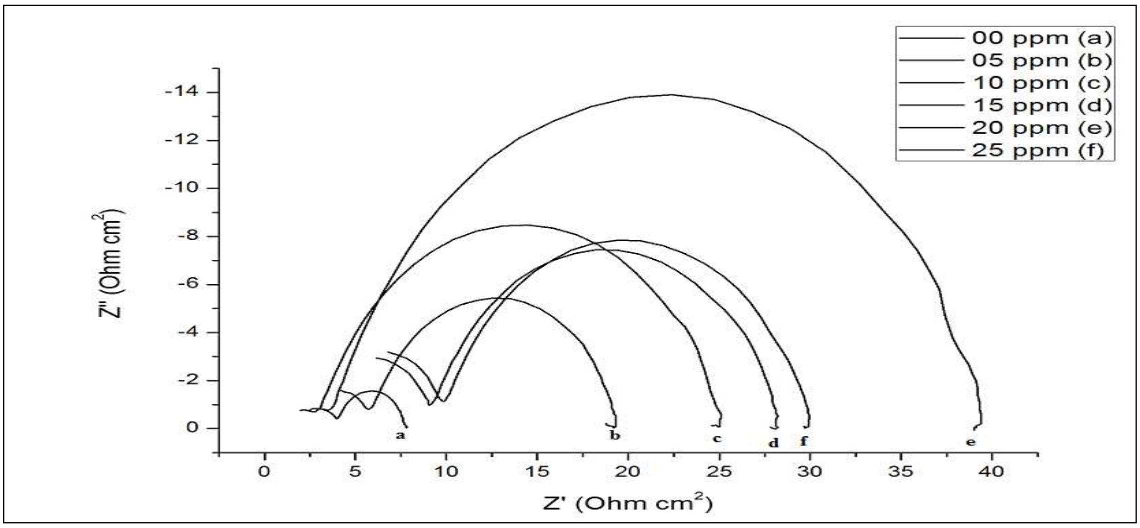

Figure. 3Nyquist plots of mild steel immersed in 1N HCl in absence and presence of different concentrations of MA leaves extract

The corrosion behavior of mild steel in 1N HCl solution of the MA was investigated by the Electrochemical Impedance spectroscopy (EIS) techniques, provide information about the kinetics of the electrode processes and simultaneously about the surface properties of the systems. The shape of impedance gives mechanistic information. Nyquist plots of mild steel in uninhibited and inhibited acid solution containing various concentrations of MA extract are presented in Figure. 3. It is followed from Figure. 3 that the impedance of the inhibited mild steel increases with increase in the inhibitor concentration and consequently the inhibition efficiency increased. The presence of a single semi - circle in the blank and for different concentrations of the inhibitor systems corresponds to the single charge transfer mechanism during dissolution of mild steel, which is unaltered by the presence of inhibitor components.

The impedance parameters derived from EIS measurements for mild steel in 1N HCl with and without inhibitors are given in Table. 3. The charge transfer resistance (R_{ct})value calculated for blank mild steel exhibited 4.170 Ωcm^2 and the double layer capacitance (C_{dl}) was 2.487x10^{-2} µF/cm^{-2}. Addition of the MA plant extract increases C_{dl}value indicating reductionof surface in homogeneity due to the adsorption of plant extract molecules.

The higher R_{ct} value obtained for higher inhibitor concentration suggests that a protective film is formed on the surface of the metal. The decreased in the C_{dl} values results from a decrease in local dielectric constant and/or an increase in the thickness of the double layer, suggested that inhibitor molecules inhibit the mild steel corrosion by adsorption at the metal/acid interface. Further, the adsorption may also due to the electronegative hetero atoms present in the organic constituents of the extract on the electropositive metal surface. All the electrochemical parameters clearly proposed that the corrosion control depends on the concentration of the inhibitor.

Table. 3 The electrochemical parameters (EIS) for mild steel corrosion rate in 1N HCl solution in different concentrations of MA leaves extract

Conc. Of MA extract(ppm)	C_{dl} (µF cm^{-2})	b_c(mV/dec)	b_a.(mV/dec)	Rct (Ω cm^2)	IE (%)
0	2.487x10^{-2}	208	168	4.170	*
5	2.013x10^{-3}	176	129	14.626	71.48
10	7.708x10^{-4}	166	86	24.659	83.08
15	1.035x10^{-3}	160	138	20.701	79.85
20	3.013x10^{-4}	143	94	38.772	89.24
25	9.334 x10^{-4}	168	111	21.523	80.62

4.5 Bode Plot

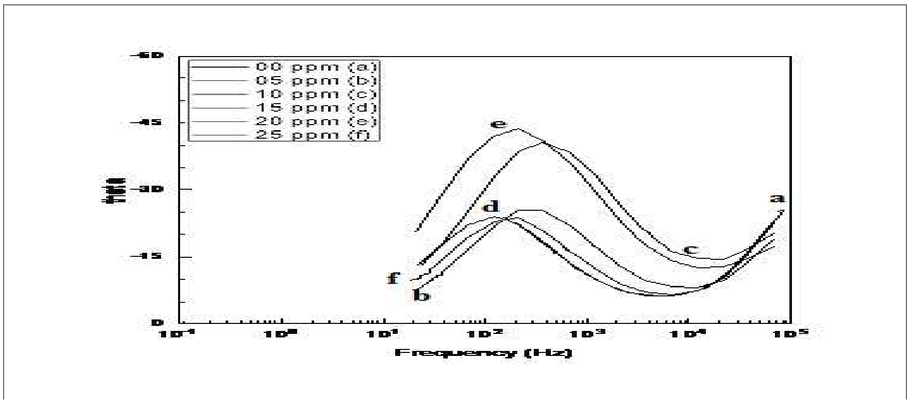

Figure. 4 Bode plots of mild steel immersed in 1N HCl in absence and presence of different concentrations of MA leaves extract

Bode plots of mild steel in uninhibited and inhibited acid solution containing various concentrations of MA extract are presented in Figure. 4. It is apparent that the mild steel specimens with MA extract showed increase in maximal phase angle value, which indicated a inhibition property on the surface mild steel. The linear portion observed in the low frequency region indicated that the diffusion process controlled the metal dissolution rate at the surface of mild steel.

4.6 Scanning Electron Microscopy (SEM)

In order to evaluate the surface morphology of the composite surface in contact with hydrochloric acid solution, a superficial analysis was carried out of the mild steel specimens with and without inhibitor in 1N HCl examined by Scanning Electron Microscopy. SEM images for the mild steel specimens exposed to 1N HCl in the absence and presence of MA extract are shown in Figure. 5(A and B). From SEM images, It can be concluded that MA leaves extract inhibited mild steel dissolution in acid by covering the surface area with protective film which has found absent in case of acid interaction with mild steel. Examination of Figure. 5A revealed that the specimen immersed in 1N HCl was rough and highly damaged due to the attack of aggressive acids. Figure. 5B clearly showed that the mild steel surface was covered with the protective layer formed by inhibitor which prevents the metal from further attack of acid medium thus inhibiting corrosion.

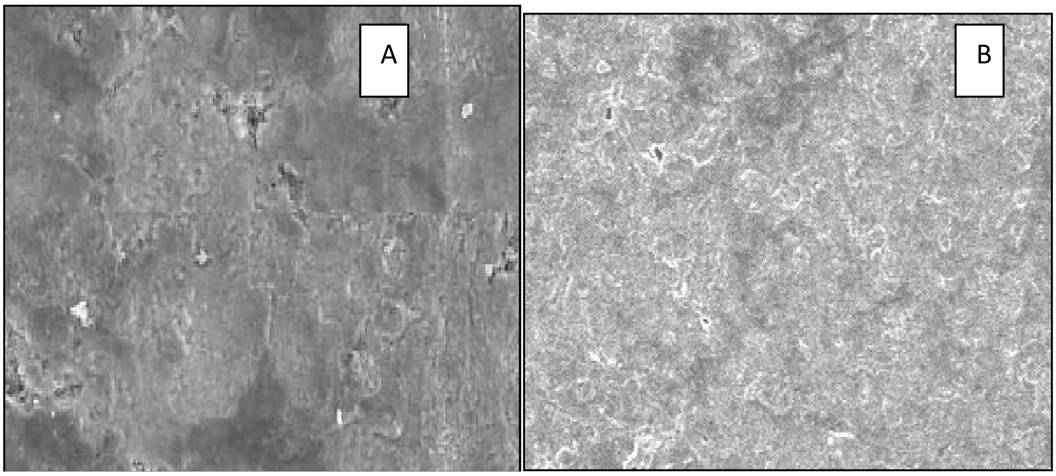

Figure. 5 SEM image of the surface of mild steel after immersion for 24 hrs in 1N HCl solution. (A) in the absence of inhibitor (B) in the presence of 20 ppm MA leaves extract

4.7 Phytochemical Screening

Phytochemical screening of the aerial parts of plant's powder (aqueous) extract was tested in order to find the presence of various chemical constituent included Alkaloids, Carbohydrates, Reducing sugars, Flavonoids, Glycosides, Saponins, Triterpenoids& Steroids, Tannins and Phenolic Compounds the results are listed in Table. 4.

Table. 4 Phytochemical screening of leaves extract of MA

S.No	Phytochemical Test	Aqueous extract of MA
1	Alkaloids	+
2	Carbohydrates	+
3	Reducing Sugars	+
4	Flavanoids	-
5	Glycosides	-
6	Saponins	+
7	Triterpenoids& Steroids	-
8	Tannins & Phenolic Compounds	-

+ is Present - is absent

4. 8. Effect of immersion time

The variation of inhibition efficiency for different concentration of plant extract of MA was listed in the Table 5. Maximum inhibition efficiency for 1N HCl was found to be 98.22% at 24 h with 25 ppm concentration of the inhibitor respectively. This behaviour may be attributed to the increase of the surface coverage by the extract, which retards the corrosion of mild steel.

Table 5. Inhibition efficiency as a various immersion time

Conc. (ppm)	IE (%)				
	1h	3h	5h	7h	24h
5	56.27	56.15	70.14	73.00	74.30
10	58.89	62.39	78.22	78.36	79.12
15	69.12	72.97	83.14	83.75	87.53
20	74.56	80.16	84.65	84.07	96.12
25	78.12	82.10	86.28	86.18	98.22

4.9. Effect of temperature

The effect of temperature on the corrosion inhibition properties of MA leaves extract was studied by exposing the mild steel to 1 N HCl containing 5, 10, 15, 20, 25ppm of the leaves extract in the temperature range of 303-323K. The data in Table. 6, indicate that the leaves extract is effective as inhibitor for mild steel in 1N HCl upto 303K and increase thereafter. The inhibition showed a maximum of 64.18 % at 303K for MA leaves extract in 1N HCl.

Table 6. IE at various Temperature

Concentration of inhibitor (ppm)	IE (%)		
	303K	313K	323K
5	40.10	34.90	22.00
10	44.50	37.57	37.09
15	52.90	50.62	49.86
20	55.26	57.89	55.66
25	64.18	58.61	56.12

4. 10. Adsorption isotherms

The results of weight loss study (Table 6) showed that the percentage of IE increases with increase in the concentration of inhibitor. This suggests that the corrosion inhibitive activity is mainly due to adsorption of various components of plant species on to the MS surface, particularly adsorption of the basic constituents like alkaloids, flavonoids, polyphenols, hydrolysis products of proteins, amine compounds which are present in the plant extract may be the reason for anticorrosion activity of the analyzed plant species. In the case of *MA (Leaves)* increase of temperature increases the IE but in most of the cases as the temperature increases the IE decreases. It suggests that the inhibition occurred through chemisorption of phytoconstituents on the Mild steel surface. The increase of temperature decreases the hydrogen evolution overvoltage that leads to the spurt in the cathodic reaction. On the other hand increase of temperature accelerates the chemisorption of the inhibitor on the metal surface. When the latter effect is predominant the final result is an increase of the inhibiting effect, which was observed in most investigated plants.

Weight loss data are quite useful in determining inhibitor adsorption characteristics. Such data are applied in construction of adsorption isotherms which give detailed information on adsorption mechanism. Well known isotherm Temkin isotherm was tested for all the data. For Temkin isotherm, surface coverage (θ) was plotted against Ln C (Fig. 6). A straight line was obtained for all the plants indicating that the green inhibitors follow Temkin isotherms.

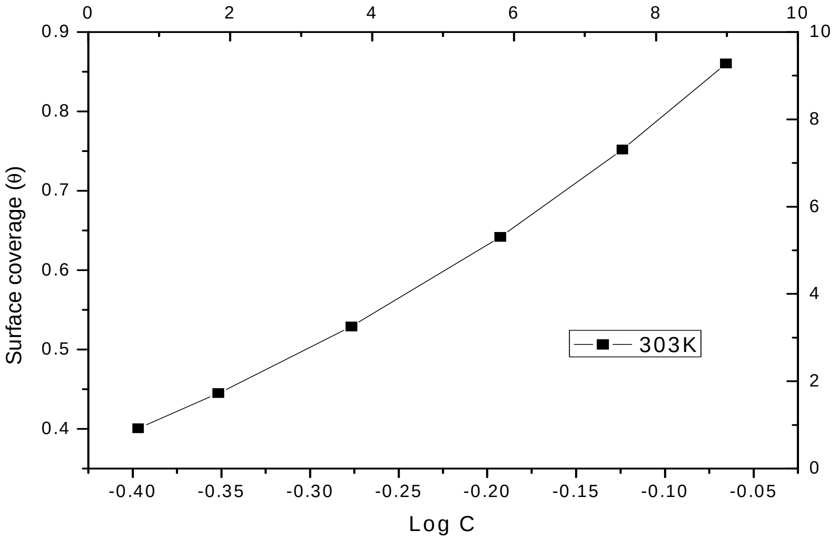

Figure.6 Temkin adsorption isotherm plot for mild steel in 1N HCl containing different concentration of the extract

5. Mechanism of inhibition

The probable mechanism of inhibition can be explained on the basis of adsorption process and the structure of the constituents present in the MA extracts. Inhibitor functions by adsorption or hydrogen bonding to the metal. This depends on the chemical composition and structure of the inhibitor, the nature of metal surface, and the properties of the medium. Structural and electronic parameters like functional group, steric and electronic effects may also be responsible for inhibition efficiency of any inhibitor that was the adsorption mechanism. The compounds have to block the active corrosion sites on the metal surface and hence the adsorption occurs by the bonding of the free electron of inhibitor with the metal. The MA extracts may constitute organic compounds containing (i) lone pair of electron present on a hetro atom (eg. N, S, P, O) (ii) pi –bonds (iii) triple bonds (eg. Cyano groups) and (iv) heterocyclic compounds such as pyridine ring, pyrrole, imidazole etc. Phytochemical analysis of MA plants extract showed the presence of alkaloids, flavonoids, saponins, tannins, glycosides and steroids. These compounds possess' hetro atoms such as – O and – N which strengthen their adsorptive property over mild steel surface. The inhibiting efficiency of these molecules may be attributed to their adsorption through the –NH, C=O, OH, COOH etc. groups and also may be due to presence of pi electrons in the rings. These organic molecules get physisorbed on the metal surface forming a protective film and hence the anti – corrosive behaviour.

CONCLUSION

The results obtained show that MA leaves extract is a good corrosion inhibitor for mild steel under acidic condition. The maximum inhibition efficiency was 90.91 %. Good agreement between the inhibition efficiencies calculated using different techniques was obtained. The adsorption of the green inhibitor onto the mild steel surface was characterized by the decrease in (i) the cathodic and anodic current densities observed in the potentiodynamic polarization curves carried out in the presence of MA leaves extract, (ii) the polarization resistance in the solution containing the inhibitor, (iii) the double capitance computed from electrochemical impedance spectroscopy experiments. It was also found that inhibitor worked as a mixed type inhibitor retarding both anodic and cathodic reactions. Surface images of the mild steel surface clearly showed that MA leaves extract inhibited corrosion of mild steel by getting adsorbed on the metal surface.

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