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STUDY ON THE EFFECT OF *MUSA ACUMINATA* FLOWER EXTRACT ON THE CORROSION INHIBITION OF MILD STEEL IN 1 N H_2SO_4

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ABSTRACT

The corrosion inhibition performance of *Musa acuminata* flower [MAN (F)] extract on mild steel in 1 N sulphuric acid solution was monitored by weight loss measurements and electrochemical technique like potentiodynamic measurement for various concentrations of the extract and in the temperature range 303 K to 353 K. The inhibition efficiency of MAN (F) extract increased with the increase of concentration but decreased with the increase in temperature. Inhibitor showed a maximum efficiency of 95.01 % at 2 % v/v extract concentration for 5 hours immersion. The polarization curves indicated that MAN (F) acts as a mixed type inhibitor. Electrochemical impedance spectroscopy studies showed the change in charge transfer capacitance with the change in concentration of the flower extract. The Temkin adsorption isotherm was tested for its fit to the experimental data. Results of SEM study on the surface morphology of mild steel in uninhibited and inhibited acid solution showed that the corrosion rate is reduced to a low value in the presence of the inhibitor. This may be due to adsorption of inhibitor molecules on the surface as a protective layer preventing acid attack. A mechanism of physical adsorption of the phytochemical components on the surface of the metal is proposed for the inhibition mechanism.

Key words: Mild steel, Sulphuric acid, Flower extract, Weight loss, Electrochemical polarization, EIS, SEM.

INTRODUCTION

Mild steels are used extensively in chemical as well as other allied industries[1]. These metal structures are often subjected to cleaning, descaling and pickling by acids, which are normally accompanied by considerable dissolution of metal as well as acid consumption[2]. Inhibitors are generally used to control metal dissolution[3]. Most corrosion inhibitors are organic compounds containing heteroatom like O, S and/or N atoms in their aromatic or long carbon chain[4]. The synthetic chemicals though are most effective corrosion inhibitive, they

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are highly toxic, costly and pose threat to the environment and make it necessary to develop environmentally acceptable and less expensive inhibitors. Natural products are good source of eco-friendly corrosion inhibitors[5]. Extracts of naturally occurring plants products contain mixture of compounds and are biodegradable in nature and at the same time offer comparable performance and cost[6].

The corrosion inhibitive properties of *Musa acuminata* flower were studied on mild steel in sulphuric acid medium. All the parts of *Musa acuminata* (Cultivar variety - Nendran), a monoecious plant of musa species have medicinal properties[7-9]. The flower extract are used to treat dysentery, diarrhoea, ulcers, diabetes and gynaecological problems[10]. It is the most nutritious herbal flower rich in vitamins, minerals, essential amino acids and an excellent source of antioxidants[11,12]. The phytochemical components of *Musa acuminata* flower have been studied and it is known to have tannins, flavonoids, saponin, alkaloid and phenols, which are responsible for inhibiting corrosion[13]. The natural flavonoids present in the flower extract exhibit antioxidative activity that stop oxidation and hence corrosion[14].

In the present investigation, the corrosion of mild steel in 1 N sulphuric acid in absence and presence of MAN (F) at 303 K – 353 K has been studied by weight loss and polarisation techniques. The adsorption characteristic of MAN (F) was studied to assess the mechanism of corrosion inhibition and the adsorption isotherm.

EXPERIMENTAL

Preparation of specimens

The commercially available mild steel was cut into coupons having dimensions of 5 x 1 x 0.2 cm with a small hole at the top end to facilitate suspension of the coupons in the test solutions. The coupons were polished using emery paper of grade 200, 400, and 600 and then washed with doubled distilled water, rinsed with acetone and dried. These plates were used for weight loss studies. Mild steel rod of 15 cm long and 5 mm diameter mounted in Teflon leaving 0.19625 cm^2 of surface for exposure to the solution was used for electrochemical studies[2].

Preparation of flower extract

The *Musa acuminata* (Nendran) flowers were collected from the farm at Thirumalayampalayam, Coimbatore. The slim, nectar-rich, tubular toothed, white flowers were separated from the purple thick, waxy hood like bract and shadow dried at room temperature. 12.5 g of flower powder were refluxed with 1 N H_2SO_4 for 3 h and left overnight to obtain the basic nutrients. The solution was filtered and filtrate made up to

250 mL to obtain 5 % stock solution, which is used in preparing different concentrations of the extract from 0.05 to 2.0 % v/v[15].

Weight loss method

Effect of concentration

The prepared mild steel coupons were immersed in 100 mL of the test solution without and with the MAN (F) extract of different concentrations for 1 h, 3 h, 5 h, 7 h, 12 h and 24 h at room temperature. After the prescribed time of immersion the coupons were removed and washed with distilled water and dried. The weight of the coupons before and after immersion was determined. Inhibition efficiency of the mild steel was then calculated[16].

Effect of temperature

The polished and pre-weighed specimens were suspended in 100 mL of the test solution without and with the addition of different concentration of the flower extract for 1 h in the temperature range of 303-353 K in the water thermostat. The specimens were removed from the test solution after 1 h and washed with distilled water, dried and weighed. The inhibition efficiency was then calculated from the weight loss[17].

Electrochemical measurements

Electrochemical experiments were carried out using computer controlled Parstat 2273. Data acquisition was performed using the Power Suite software and analyzed using ZsimpWin software (version 3.21). A three electrode set up with Platinum foil as auxiliary electrode and a saturated calomel electrode as the reference electrode was employed. The mild steel rod with surface prepared as described in weight loss experimental method served as the working electrode. The measurements were carried out in the frequency range of $10^6 – 10^{-2}$ Hz at the open circuit potential by superimposing a sinusoidal AC signal of small amplitude, 10 mV after immersion for 30 min in the corrosive medium. The double layer capacitance (C_{dl}) and charge transfer resistance (R_{ct}) were obtained from the Nyquist plots. The potentiodynamic polarization curves were recorded using the same cell setup employed for the impedance measurements[18]. The potentials were swept at the rate of 1.66 mVs^{-1}.

SEM analysis

The surface microstructure of specimens after 1 hour immersion in 1 N H_2SO_4 and an optimum concentration of inhibitor 2% v/v was studied to understand the changes that occur during the corrosion of mild steel in the presence and absence of flower extract, using JEOL Scanning Electron Microscope – Model JSM 6360.

RESULTS AND DISCUSSION

Effect of concentration

The weight loss method was carried out with the concentration ranging from 0.05% to 2.00% v/v. The weight loss data are listed in Table 1. The results show that with rise in concentration of MAN (F) extract, the inhibition efficiency increased[19]. At optimum concentration of 2% v/v it has a maximum inhibition efficiency of 95.01%. This result indicated that MAN (F) act as an excellent corrosion inhibitor. This is attributed to the absorption of nutrients of the flower on the surface of mild steel which make a barrier for mass and charge transfer and prevent further corrosion[20].

Effect of immersion time

Table 1 and Fig. 1 show the effect of immersion time on corrosion of mild steel. The MAN (F) extract has maximum efficiency of 95.01 % at 5 h immersion time indicating the increase of adsorbed inhibitor molecules on the mild steel surface with time. The inhibition efficiency was found to decrease from 95.01 % to 91.28 % as immersion time was increased from 5 h to 24 h. This may be because, prolonged immersion may result in desorption of the inhibitor molecules from the mild steel surface[21].

Table 1: Inhibition efficiency of mild steel in 1 N H_2SO_4 in the presence of MAN (F) extract

Conc. of extract in v/v %	Percentage inhibition efficiency (IE %)					
	Immersion time in hours					
	1	3	5	7	12	24
0.05	68.10	71.81	66.67	64.05	53.95	45.23
0.10	78.08	81.16	79.68	74.60	68.18	60.07
0.50	87.48	89.41	88.07	87.94	85.98	83.70
1.00	88.45	90.65	91.11	90.70	89.49	86.72
1.50	90.22	91.89	92.60	91.20	91.84	90.95
2.00	91.98	94.70	95.01	94.08	92.26	91.28

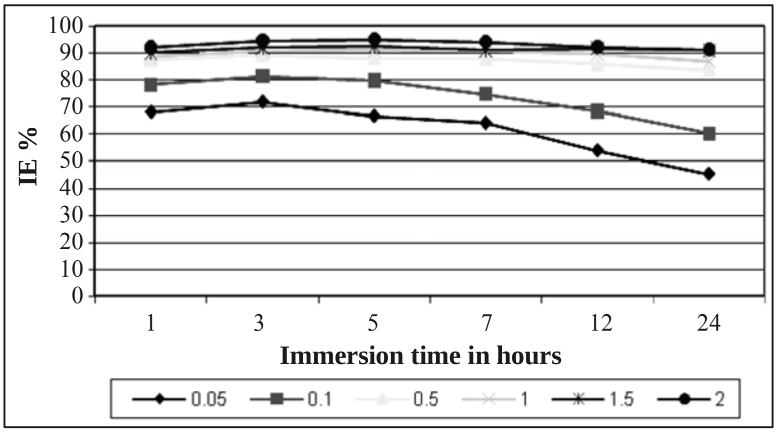

Fig. 1: Influence of immersion time on IE % of MAN (F) extract in 1 N H_2SO_4

Effect of temperature

The effect of temperature on the corrosion inhibition properties of flower extract was studied by exposing the mild steel to 1 N H_2SO_4 containing 0.05, 0.10, 0.50, 1.00, 1.50, 2.00 % v/v of the flower extract in the temperature range of 303-353 K. The data in Table 2 indicate that the flower extract is effective as inhibitor for mild steel in 1 N H_2SO_4 up to 313 K and decreases thereafter. A maximum inhibition of 93.60 % at 313 K was observed for 2 % v/v flower extract in 1 N H_2SO_4 (Fig. 2).

Table 2: Effect of temperature on mild steel corrosion in 1 N H_2SO_4 in presence of MAN (F) extract

Conc. % v/v	Percentage inhibition efficiency (IE %)					
	303 K	313 K	323 K	333 K	343 K	353 K
0.05	68.10	73.21	65.67	62.53	48.50	34.19
0.10	78.08	78.69	79.52	69.90	63.45	38.86
0.50	87.48	88.19	88.61	85.32	80.65	66.78
1.00	88.45	91.49	89.07	88.33	86.05	72.20
1.50	90.22	92.62	90.78	89.65	88.52	77.76
2.00	91.98	93.60	92.91	91.24	89.03	79.96

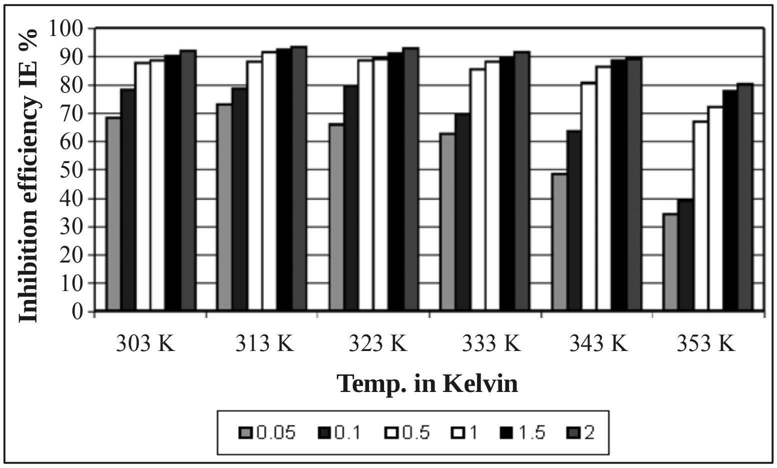

Fig. 2: Effect of temperature on inhibition efficiency of mild steel in 1 N H_2SO_4 in presence of MAN (F) extract

An increase in temperature influences the adsorption equilibra and kinetics. Fig. 3 shows the Arrhenius plots for corrosion of mild steel in H_2SO_4 medium in the presence and absence of the inhibitor. From the slopes of the plots, the activation energy values were calculated. Table 3 shows that the values of activation energy increase in the presence of the inhibitor than that in the blank. The higher value of activation energy in the presence of inhibitor leads to a conclusion that the inhibitor has adsorbed on the surface by physisorption process[22].

Table 3: Activation energy values of mild steel corrosion in 1 N H_2SO_4 in absence and presence of MAN (F) extract

Conc. % v/v	E_a (J/mol)
Blank	-44116.39
0.05	-58260.77
0.10	-61853.15
0.50	-60466.01
1.00	-58720.95
1.50	-57755.18
2.00	-59902.51

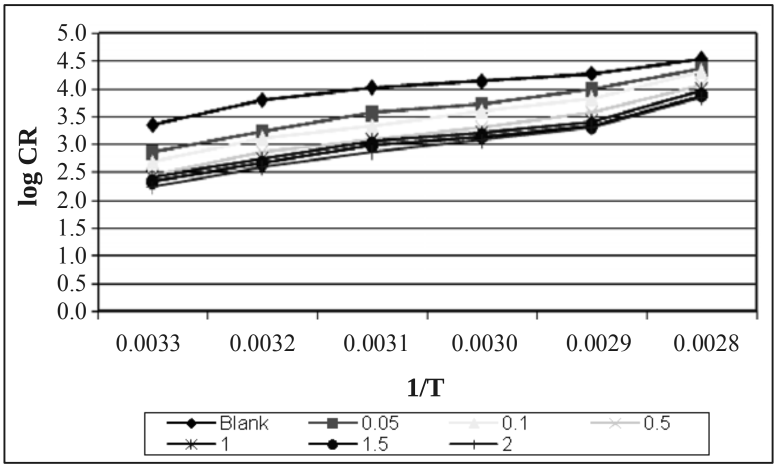

Fig. 3: Arrhenius plots of mild steel corrosion in absence and presence of MAN (F) extract in 1 N H_2SO_4

Potentiodynamic polarisation results

The various electrochemical parameters calculated from the Tafel plot of Fig. 4 are given in Table 4. The lower corrosion current density (I_{corr}) values in the presence of inhibitor without causing significant changes in corrosion potential (E_{corr}) suggests that the compound is mixed type inhibitor and are absorbed on the surface blocking the corrosion process[23]. In all concentrations, b_c is greater than b_a suggesting that though inhibition is under mixed control, the effect of the inhibitor on the anodic polarization is less pronounced than on the cathodic polarization[24,25].

Table 4: Potentiodynamic polarization parameters for mild steel in 1 N H_2SO_4 in the absence and presence of MAN (F) extract

Concentration of inhibitor (% v/v)	$-E_{corr}$ V	I_{corr} mA mp cm^2	b_a mV/dec	b_c mV/dec.	R_p Ohm cm^2	% Inhibition efficiency	
						Tafel	Linear
Blank	0.491	1005.00	81.30	180.69	3.30	-	-
0.05	0.495	902.80	78.19	185.90	4.39	10.17	24.83
0.50	0.488	730.90	69.31	188.41	6.98	27.27	52.72
1.00	0.484	486.40	61.11	183.09	10.28	51.60	67.90
2.00	0.483	419.20	67.00	182.14	13.90	58.29	76.26

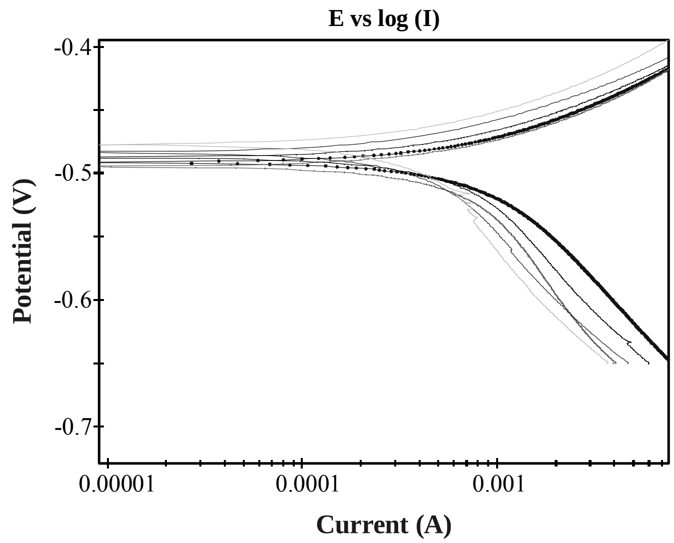

Fig. 4: Tafel Polarization parameters for mild steel in 1 N H_2SO_4 in the presence and absence of MAN (F)

Electrochemical impedance spectroscopy results

Fig. 5 shows the Nyquist plot obtained for mild steel in 1 N H_2SO_4 in the presence of various concentrations of the inhibitor. The impedance parameters from Nyquist plot are given in Table 5. Results show that as the concentration of inhibitor increases C_{dl} values decrease, which can result from an increase in thickness of electrical double layer, suggesting that the inhibitor molecule function by adsorbing at the metal-solution interface[26].

Table 5: Impedance parameters for mild steel in the absence and presence of MAN (F) extract in 1 N H_2SO_4

Concentration of inhibitor (% v/v)	C_{dl} (μF cm^{-2})	R_{ct} Ohm cm^2	Inhibitor efficiency	
			C_{dl}	R_{ct}
Blank	11.07 x 10^{-5}	18.25	-	-
0.05	8.849 x 10^{-5}	22.83	20.06	20.06
0.50	5.747 x 10^{-5}	29.25	48.09	37.61
1.00	4.100 x 10^{-5}	49.28	62.96	62.97
2.00	3.107 x 10^{-5}	65.02	71.93	71.93

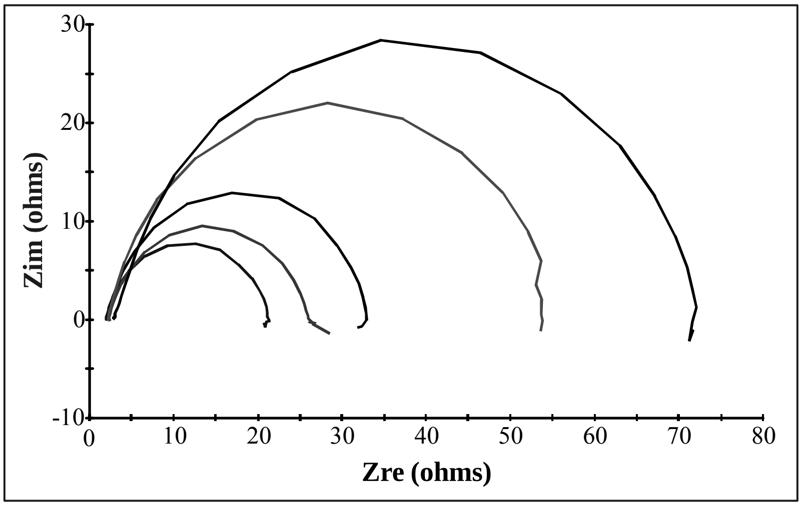

Fig. 5: Nyquist plot-Mild steel in 1 N H_2SO_4 in the absence and presence of MAN (F)

SEM analysis

Examination of SEM image (Fig. 6) reveal that the specimen immersed in 1 N H_2SO_4 was highly damaged. Fig. 7 clearly showed the surface of mild steel being highly covered with protective layer formed by the adsorption of nutrients present in the flower extract, which prevents the metal from further attack by acid and thus inhibiting corrosion[26, 27].

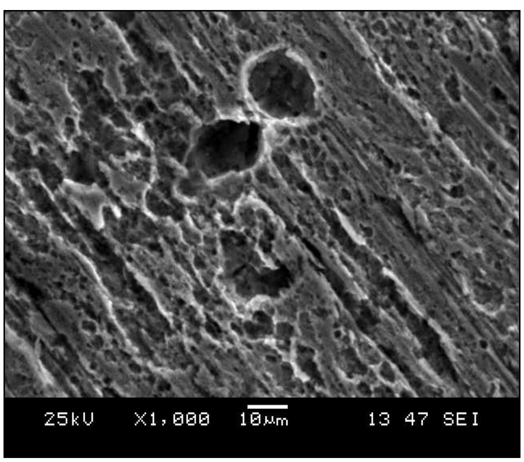

Fig. 6: Mild steel surface after immersion in 1 N H_2SO_4 for 1 hour

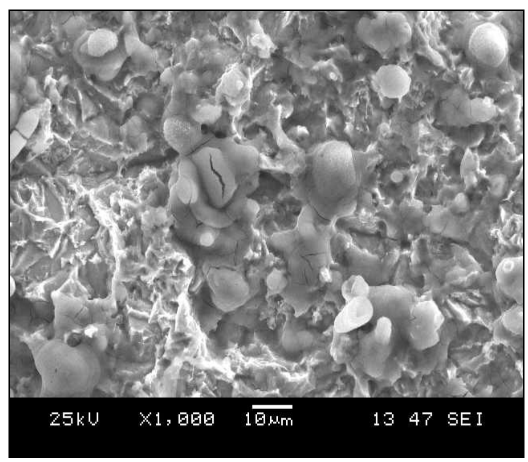

Fig. 7: Mild steel surface after immersion in 1 N H_2SO_4 containing 2% v/v MAN (F) extract for 1 hour

Mechanism of corrosion inhibitor

The mechanism of inhibition can be understood by the mode of adsorption of the flower nutrients on the mild steel surface. The surface coverage θ, (θ = IE / 100) values calculated using weight loss method for different concentrations of inhibitors were tested graphically by fitting to Temkin isotherm. A plot of θ against log C for different concentration (Fig. 8) shows a straight line indicating that the extract follow the Temkin adsorption isotherm[28].

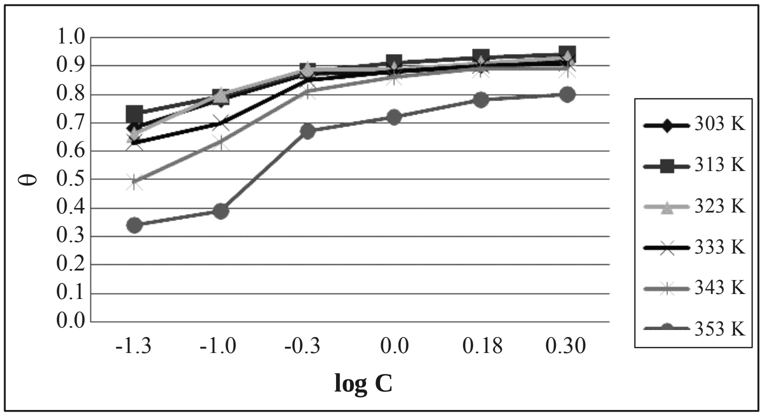

Fig. 8: Temkin adsorption isotherm for MAN (F) on mild steel in 1 N H_2SO_4 at different temperatures

Musa acuminata flower comprises of tannin, flavonoid, saponin, alkaloid, phenol and amino acids. Most of these organic compounds possess heteroatom such as O- and N-, which strengthen the adsorptive property on the metal surface and hence the anti-corrosive behaviour[29]. Move over nitrogen containing compounds show their best efficiencies in sulphuric acid. Thus, it can be suggested that the high inhibition efficiency of MAN (F) may be due to active nutrients of MAN (F) containing oxygen and nitrogen[30].

CONCLUSION

The adsorption and inhibition efficiency of *Musa acuminata* flower extract on mild steel in 1 N H_2SO_4 was studied using weight loss and electrochemical methods: the following conclusions were drawn from the present studies.

The inhibitor efficiency of MAN (F) extracts increases with an increase in inhibitor concentration but decreased with temperature. The maximum efficiency 95.01% was observed at an optimum concentration of 2% v/v. The potentiodynamic polarization curves recorded reveal that MAN (F) acts as a mixed type of inhibitor. The adsorption of MAN (F) extract onto the surface follows Temkin adsorption. Surface studies involving SEM confirmed the efficiency of the flower extract as corrosion inhibitor for mild steel.

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