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RESEARCH ARTICLE

Corrosion Inhibition and Adsorption Properties of African marigold for the Corrosion of Mild Steel in Hydrochloric acid

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ABSTRACT:

Inhibitive and adsorption properties of acid extract of flowers of African marigold (AMF) for the corrosion of mild steel in hydrochloric acid medium was studied using weight loss and electrochemical studies. The results revealed that acid extract of African marigold flowers inhibited the corrosion of mild steel. The inhibition efficiency of the extract increases with increase in concentration of the extract, exposure time and temperature of the medium. The maximum inhibition efficiency of 97% was obtained at 0.3%v/v concentration of the extract at room temperature. Potentiodynamic polarization study clearly reveals that the extract controls both anodic as well as cathodic reactions. Double layer capacitance (Cdl) and charge transfer resistance (Rct) values were derived from Nyquist plots. The adsorption of extract components on the mild steel surface was found to follow the Langmuir adsorption isotherm. The energy of activation, enthalpy of activation and entropy of activation for the dissolution process were determined and discussed. A mechanism of adsorption of the flower components on the surface of the metal was proposed for the inhibition behavior on the basis of FTIR study.

KEYWORDS: Corrosion rate, inhibition efficiency, Potentiodynamic polarization, FTIR, adsorption isotherm.

INTRODUCTION:

Mild steel is used as a structural material for reaction vessels, pipes, tank etc. which are known to corrode invariably in contact with various solvents and acids. It is necessary to adopt appropriate means and ways to reduce the losses due to corrosion, from the view point of nation's economy and financial implications of corrosion hazard. In order to prevent or minimize corrosion, inhibitors are used especially in industries. Organic, inorganic, or a mixture of both can inhibit corrosion by either chemisorption on the metal surface or reacting with metal ions and forming a barrier-type precipitate on its surface (1-8).

Because of the toxic nature and /or high cost of some chemicals currently in use as inhibitors, it is necessary to develop environmentally acceptable and inexpensive ones. Natural products can be considered as a good source for this purpose. The different parts of some plants such as *Azadirachta Indica* (9), *Colocasia esculenta*(10), *Murraya Koenigh* (11), *Rauvolfia Serpentina* (12), *Hybiseus Syriacus* Linn (13), *Foeniculum Vulgare* (14), *Citrus aurantiifolia* (15) have been found to be good corrosion inhibitors for many metals and alloys.

As a contribution to the current interest on environmentally friendly corrosion inhibitors, the present study investigates the inhibiting effect of flowers extract of *African marigold* on mild steel in hydrochloric acid medium.

Experimental Procedures:

Preparation of specimen:

Mild steel specimens of dimension 1×5×1cm were cut from a sheet of commercial cold rolled mild steel and mechanically polished to a mirror finish, degreased with acetone and then rinsed with distilled water, dried, stored in desiccators and used for the weight loss studies.

Preparation of extract:

The Botanical name of African marigold is *Tagetes erecta*, belongs to Compositae family. The flowers of African marigold were collected shade dried, crushed and powdered. From this powder, 25g was taken in a RB flask, refluxed with 500ml of acid for 3h and kept it overnight. The next day, it was filtered and made up to 500ml in a standard flask with the same acid. This solution was stored as stock solution and used for further studies.

Weight loss studies:

Weighed test pieces were separately and fully immersed for different periods (1h, 3h, 5h, 7h, 12h and 24h) in each of the beaker containing the hydrochloric acid without and

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with the inhibitor (0.001,0.005,0.01,0.05,0.1,0.2 and 0.3%V/V). Each of the test specimens were removed, neutralized with sodium bicarbonate, rinsed with water, dried and re-weighed.

The corrosion rate and inhibition efficiency were calculated using the following equations

$$C.R = \frac{534 \times W \times 1000}{DAT}$$

Where C.R is the corrosion rate in mpy

W is the weight loss of the substance in grams, D is the density of mild steel in g/cm^3, A is the area of the specimen in inch

$$I.E\,(\%) = \frac{C.R - C.R\,(inh)}{C.R} \times 100$$

T is the time of immersion in hours

Where C.R – is the corrosion rate without inhibitor
C.R (inh) – is the corrosion rate with inhibitor.

Potentiodynamic polarization measurements:

Potentiodynamic polarization and impedance measurements were carried out using an electrochemical instrument (model SOLATRON -1280B). A platinum foil was used as an auxiliary electrode, a saturated calomel electrode was used as reference electrode and mild steel was used as the working electrode. All the experiments were carried out at room temperature (30° ± 2° C) and a scan rate of 10mV/sec at OCP. The polarization curves were obtained after immersion of the electrode of the solution until a steady state was reached.

RESULTS AND DISCUSSION:

Weight loss method:

Effect of concentration

The inhibition efficiency was obtained from weight loss a study at different concentration of the extract at room temperature was given in table (1). It has been observed from the table that the inhibition efficiency increased with increase in extract concentrations. The maximum inhibition efficiency is found to be obtained at **0.3%V/V** of concentration of extract. The increase in inhibition efficiency with increase in concentration of the extract may be due to the adsorption of active constituents present in the extract on the mild steel surface.

Table: 1 Inhibition efficiency of Mild Steel in 1M HCl in the presence of extract (AMF)

Inhibitor Concentration V/V (%)	Inhibition Efficiency (%)				
	1h	3h	5h	7h	24h
0.001	13	29	57	73	88
0.005	41	44	67	75	92
0.01	49	64	72	77	93
0.05	53	70	78	82	95
0.1	64	74	82	82	96
0.2	73	76	84	85	96
0.3	80	80	85	88	97

Effect of immersion time

From the figure (1), it is clear that the inhibition efficiency of the extract increased with increase in immersion time. The increased inhibition efficiency with exposure time may be attributed to the increase in the surface coverage by the adsorption of phytochemical constituents of the plant extracts. It may also be due to the stability of the inhibitor in the 1M HCl leading to strong adsorption of the components present in the extracts. The optimum period of immersion to get the maximum inhibition is 24h for 0.3%V/V concentration of the extract.

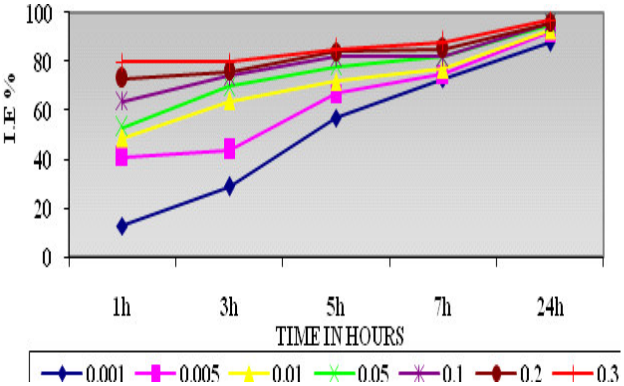


Figure: 1 Effect of immersion time on the dissolution of mild steel in HCl in the presence of extract

Effect of temperature

The influence of temperature on the inhibition efficiency of the extract is given in table (2). The inhibition efficiency of the extract increases with increase in temperature from 30° to 50°C. Beyond that, inhibition efficiency decreases with increase in temperature (ie.) from 50° to 70°C. The decrease in inhibition efficiency with temperature may be attributed to desorption of the active constituents from the metal surface at high temperature. Hence, the optimum temperature for maximum inhibition efficiency is 323K.

Table: 2 Influence of temperature on the inhibition efficiency of mild steel in HCl medium

Inhibitor Concentration V/V (%)	Inhibition efficiency (%)				
	303K	313K	323K	333K	343K
0.001	29	30	32	11	13
0.005	38	39	41	26	26
0.01	49	54	55	39	41
0.05	53	66	68	52	57
0.1	64	75	78	63	63
0.2	73	83	88	74	76
0.3	80	90	93	84	85

Activation Parameters

The effect of temperature on the dissolution of iron and its mechanism in acid media was investigated by gravimetric measurements at different temperatures (30ºC to 70ºC) for an hour immersion period in the absence and presence of various concentrations of extract. The activation parameters for the system were calculated from Arrhenius equation (1) and transition state equation (i.e) Eyring equation (2).

Log (C.R) = log A –E_a / RT ---------------- (1)

Arrhenius equation

Where A is a pre-exponential factor E_a is the apparent activation energy. A plot of log (C.R) against 1/T gives straight line of slope of E_a/2.303*R. From the slope of the straight line the activation energy was calculated

The alternative formulation of the Arrhenius equation is called the transition state equation (Eyring equation)

Log (C.R/T) = {log R/N_Ah + ΔS*/2.303*R} - ΔH*/2.303RT ------- (2)

Where h is Planck's constant, N_A is Avogadro's number, R is the universal gas constant, ΔH* is the change in enthalpy and ΔS* is the change in entropy for the corrosion process

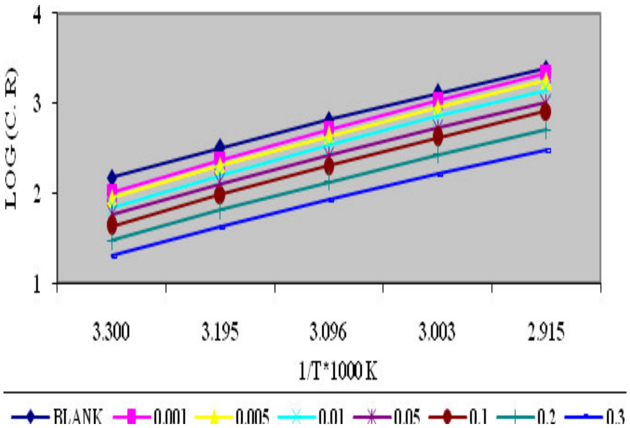
Energy of activation:

The plot log (C.R) against (1/T) gave straight lines with slope –E_a/RT figure (2). From the slope, activation energy of the metal-dissolution process was calculated and the values were given in tables (3).

Table: 3 Activation parameters for the dissolution of mild steel in HCl

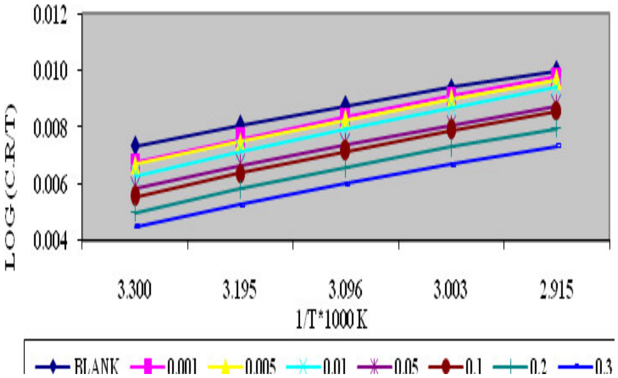
Concentration of the inhibitor (%V/V)	$-E_a$ KJ / mol	$-\Delta H_a$ KJ / mol	ΔS_a J/mol/K
Blank	59.99	132.39	62.97
0.001	65.5	151.49	62.92
0.005	64.64	150.07	62.93
0.01	64.83	152.98	62.92
0.05	61.19	142.89	62.96
0.1	63.48	151.86	62.94
0.2	61.11	147.52	62.96
0.3	58.42	142.52	62.99

From the table it can be inferred that the value of apparent activation energy E_a for 1M HCl without the inhibitor (~ 59 KJ/mole) agrees well with literature data on E_a for iron and steel in hydrochloric acid, which are in the range 58 – 100 KJ/mole (16,17,18 and 19). The addition of extracts modified the values of the apparent activation energies. This modification may be attributed to the change in the mechanism of the corrosion process in the presence of adsorbed inhibitor molecules. The average value of E_a in the presence of inhibitors in 1M HCl was 62.74 KJ/mole for extract.



Enthalpy and entropy of activation

The plot of log (C.R/T) against 1/T gave straight lines as shown in figure (3). The slope of these lines equal to -ΔH_a/2.303*R and the intercept is given by log RT/N_Ah + (-ΔS_a/2.303* R), from which the values of ΔS_a and ΔH_a were calculated and the values are given in table (3).



The negative value of enthalpy of activation showed that the metal dissolution process is accompanied by the release of higher amount of heat energy (i.e) an exothermic process. In general, an endothermic process ($\Delta H_a > 0$) is attributed unequivocally to chemisorption, while an exothermic process ($\Delta H_a < 0$) may involve either physisorption or chemisorption or a mixture of both the processes (20). In the present study, $\Delta H_a < 0$ is in conformity with Ali *et.al* findings. The entropy of activation ΔS_a in the absence and presence of extracts were large and positive. This implies that the activated complex in the rate determining step represents the dissociation rather than association step, meaning that an increase in disordering takes place on going from reactants to the activated complex.

ADSORPTION ISOTHERM

A useful method that assists in the understanding of the mechanism of organo-electrochemical reactions in the adsorption processes is the adsorption isotherm. Adsorption provides information about the interaction among the adsorbed molecules themselves as well as their interaction with the electrode surface. Attempts were made to fit θ values to various isotherms including Langmuir, Temkin, Freundlich, Frumkin, Bockris-Swinkles and Bockris, Devanathan and Muller isotherm.

Langmuir adsorption isotherm

Langmuir adsorption isotherm has been conveniently used to study inhibitor-metal interaction. Assuming that the percentage area covered by the inhibitor is directly related to the retardation in corrosion rate. The inhibitors should obey the Langmuir adsorption isotherm in which log (θ/1-θ) is a linear function of log concentration, according to the equation

$$\log (\theta/1-\theta) = \log C + \log K$$

Where, C is the bulk concentration and K is the equilibrium constant. It is observed that the plots were linear (Figure.4) and the slopes were never unity (Table 4), contrary to what is expected for ideal Langmuir adsorption isotherm equation. During the derivation of Langmuir equation, it is assumed that there are no intermolecular interactions between the adsorbate. However molecules having polar atoms (or) groups which are adsorbed on the metal surface may interact by mutual repulsion (or) attraction and this may be advocated as the reason for the departure of the slope values from unity. Similar results were obtained by many authors (21, 22 and 23).

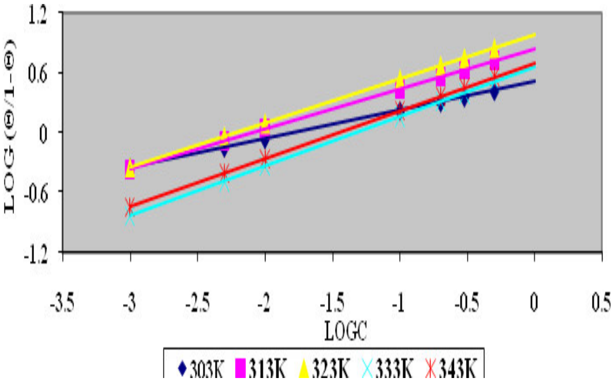


Figure: 4 Langmuir Adsorption isotherm of mild steel in 1M HCl in presence of extract

Table: 4 Regression Analysis - Langmuir adsorption isotherm

	1M HCl				
	303K	313K	323K	333K	343K
Slope	0.29	0.40	0.44	0.48	0.48
Intercept	0.50	0.84	0.97	0.66	0.70
Corr.coeff.	0.84	0.87	0.85	0.89	0.91

Potentiodynamic polarization measurement:

A Potentiodynamic anodic and cathodic polarization scan was carried out at room temperature. The various electrochemical parameters calculated from the Tafel plot is given in table (5) and the plot is shown in figure (5). The lower corrosion current density (I_{corr}) values in the presence of extract without causing significant changes in corrosion potential (E_{corr}) suggests that it is mixed type inhibitor. The changes in values of cathodic Tafel slope b_c with inhibitor concentrations from 158 to 99mV dec^{-1} and the anodic Tafel slope b_a from 212 to126 mV dec^{-1} indicates that the influence of extract both on the kinetics of hydrogen evolution and reduction of the metal dissolution along with hydrogen evolution.

Table: 5 Electrochemical parameters for mild steel in 1M HCl in presence of AMF extract

Inhibitor concentration V/V (%)	$-E_{corr}$ mV	I_{corr} µA cm^{-2}	b_a mV/dec	b_c mV/dec	I.E (%)
Blank	504	5.53	212	158	-
0.001	502	5.38	215	172	2.7
0.005	502	5.36	213	166	3.0
0.01	502	4.10	190	146	25.85
0.05	499	2.52	170	124	54.43
0.1	498	2.03	166	119	63.29
0.2	494	1.01	134	103	81.74
0.3	496	0.79	126	99	85.71

Potentiodynamic polarization of mild steel in 1M HCl in presence of AMF extract

Impedance measurement

The corrosion behaviour of mild steel in 1M HCl in the absence and presence of extracts was investigated by the electrochemical impedance spectroscopy at room temperature (30ºC). Nyquist plots of mild steel in 1M HCl containing various concentrations of extract TEF is given in figure (6) and the characteristic parameters associated to the impedance diagrams like charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) and calculated values of I.E (%) from the R_{ct} values are given in table (6).

It can be seen from the figure that, the Nyquist plot contains depressed semicircles with centres under the real axis, whose size increases with the extracts concentration, indicating charge transfer process is mainly controlling the corrosion of mild steel. Such behaviour is characteristic of solid electrodes and is often referred to as frequency dispersion. This has been attributed to roughness and other in-homogeneities of the solid surface.

From the table, it was observed that the R_{ct} values increases with increasing concentrations of the extracts. On the other hand, the value of C_{dl} decreases with an increase in the inhibitor concentration. This situation is due to an increase in the surface coverage by the inhibitor, which led to an increase in the inhibition efficiency.The decrease in the C_{dl} and the corresponding increase in R_{ct}, suggested that the extract molecules function by adsorption at the metal/solution interface. Thus the change in C_{dl} value is caused by the gradual replacement of water molecules by the adsorption of the organic molecules on the metal surface, decreasing the extent of metal dissolution.

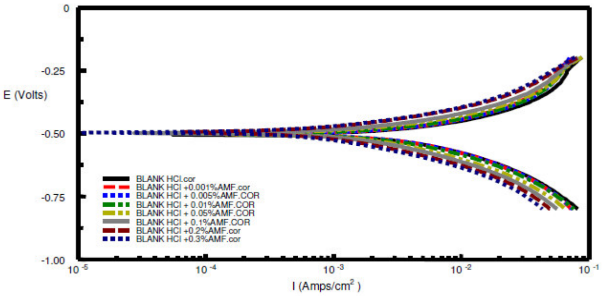


Figure: 5 Potentiodynamic polarization of mild steel in 1M HCl in presence of AMF extract

Table: 6 Impedance parameters for the corrosion of mild steel in 1M HCl

Inhibitor Concentration V/V (%)	R_{ct} (Ω cm^2)	C_{dl} (µF/cm^2)	I.E (%)
Blank	15.14	149.18	-
0.001	18.76	141.13	19.30
0.005	22.63	134.35	33.09
0.01	53.84	114.33	71.87
0.05	61.24	107.03	75.27
0.1	78.41	94.82	80.69
0.2	84.73	79.14	82.13
0.3	94.76	78.12	84.02

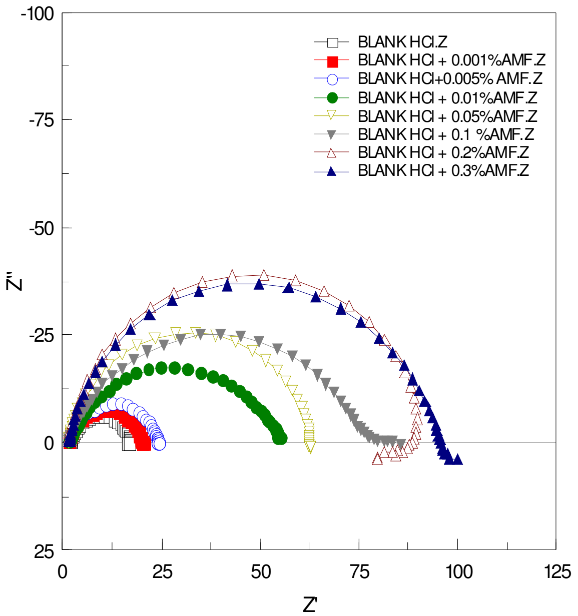


Figure: 6 Nyquist Plot of mild steel in 1M HCl

It can be seen from the figure that, the Nyquist plot contains depressed semicircles with centres under the real axis, whose size increases with the extracts concentration, indicating charge transfer process is mainly controlling the corrosion of mild steel. Such behaviour is characteristic of solid electrodes and is often referred to as frequency dispersion. This has been attributed to roughness and other in-homogeneities of the solid surface.

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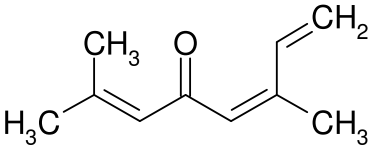
SURFACE STUDIES

FTIR spectra

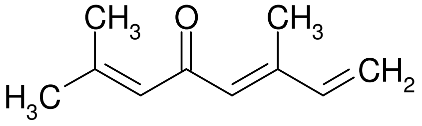
The FTIR spectra of the film formed on the surface of mild steel in the HCl solution containing the AMF extract is given in figure: 7. The broad band at about 3500cm^{-1}can be assigned to the presence of intermolecular hydrogen bond, stretching mode of an O-H and /or N-H. Monosubstituted alkenes, that is, vinyl group and simple open chain, secondary amides, absorb near 1640cm^{-1}**.** The C-O stretching vibrations produce a band in the 1260-1000cm^{-1} (24). Therefore, it is evident that the extracted organic compounds are stable in 1M HCl medium. Further, the presence of these organic compounds was identified by the various phytochemical constituents screening tests. Variation in the FTIR spectra of the film formed on the metal surface in acid in the presence of extracts (0.3% V/V), may be due to the strong physical adsorption of the constituents present in the extract leading to chemisorption.

Mechanism of corrosion inhibition:

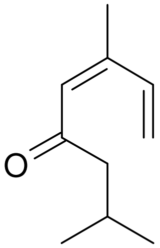
Inhibition of corrosion of mild steel in acid medium by extract may be explained on the basis of adsorption. The flowers of *African marigold* (AMF) were analyzed by a combination of GC and GC/MS. Thirty four components were identified (25, 26, 27 & 28). The main characterized constituents were β-caryophyllene (35.2%), terpinolene (6.3%), and (E)-ocimenone (9.8%), (Z) β-ocimene (13.7%), piperitenone (2.6%), Tagetone (6.2%) and limonene (2.5%).



(Z)-Ocimenone



(E)-Ocimenone



(Z)-tagetone

The constituents present in the extract inhibit corrosion by controlling both the anodic and cathodic reactions. In acidic solutions, the components may exist as protonated species. These protonated species adsorb on the cathodic sites of mild steel and decrease the evolution of hydrogen. The adsorption on anodic sites may occur through π-electrons and lone pair of electrons of oxygen atom which may decrease anodic dissolution of mild steel.

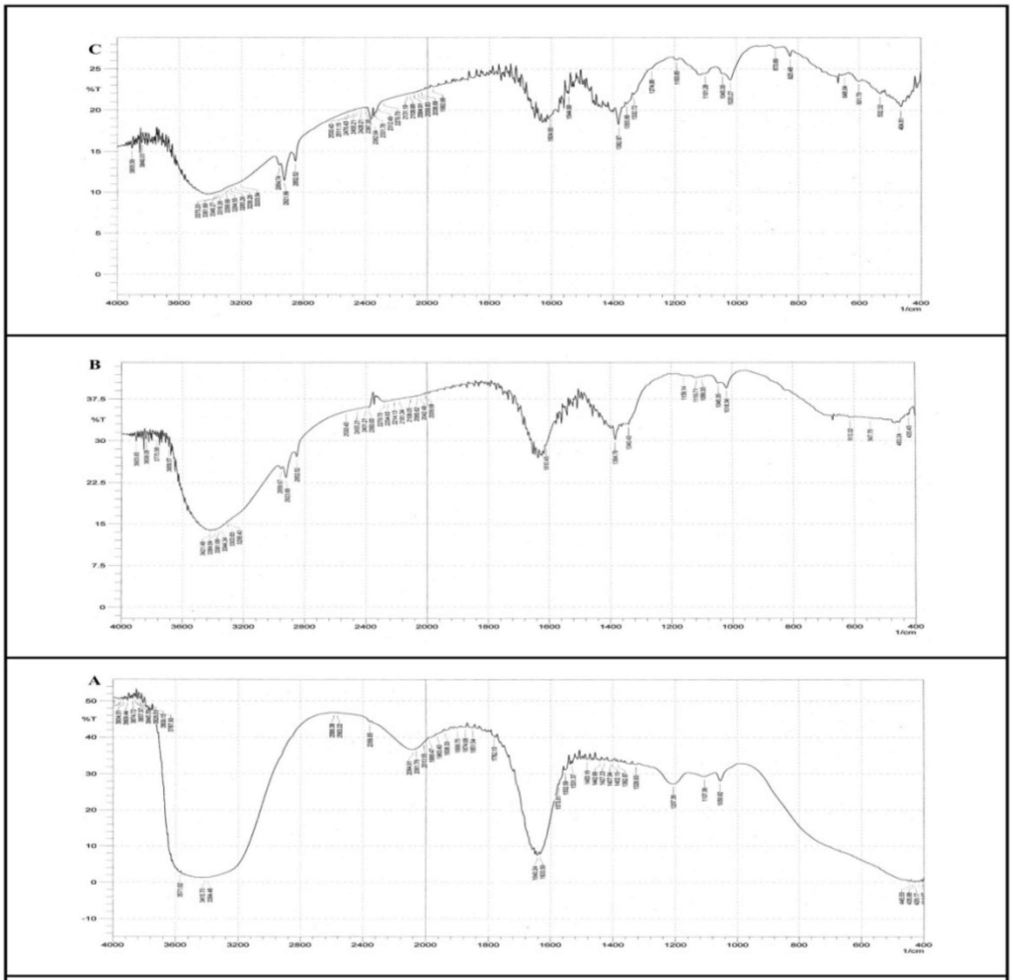


Figure : 4.106 FTIR spectra of AMF extract (A) and for the film formed on the mild steel immersed in test solutions (B) and (C)

A. Pure AMF extract
B. HCl + 0.3% AMF extract
C. H_2SO_4 + 0.3% AMF extract

FTIR spectra of AMF extract (A) and film formed on the mild steel in HCl solution (B)

CONCLUSION:

The conclusions of the above study are

- The plant extract AMF acts as a good inhibitor in HCl acid medium.
- The inhibition efficiency of the extract increases with increase in extract concentration, immersion time and temperature.
- The adsorption of the components of the extract on the mild steel obeys Langmuir adsorption isotherm.
- Potentiodynamic polarization study shows that it control both the cathodic and anodic sites of the metal surface, thus acts as a mixed type inhibitor.
- Electrochemical impedance spectroscopy shows that R_{ct} values increases, while C_{dl} values decreases in the presence of extract.
- FTIR study confirms the adsorption of the components present in the flower extract of African marigold on the mild steel through π-electrons and lone pair of electrons present in the oxygen atom.

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