International Journal Of
Recent Scientific Research
ISSN: 0976-3031
Volume: 7(4) April -2016

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THE OFFICIAL PUBLICATION OF
INTERNATIONAL JOURNAL OF RECENT SCIENTIFIC RESEARCH (IJRSR)
http://www.recentscientific.com/ recentscientific@gmail.com

ISSN: 0976-3031

Available Online at http://www.recentscientific.com

International Journal of Recent Scientific Research
Vol. 7, Issue, 4, pp. 10189-10195, April, 2016

International Journal of Recent Scientific Research

Research Article

SPIROPYRROLIDINE DERIVATIVES AS CORROSION INHIBITOR FOR MILD STEEL IN 1M HCl

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ARTICLE INFO

Article History:

Received 05th January, 2016
Received in revised form 08th February, 2016
Accepted 10th March, 2016
Published online 28st April, 2016

Keywords:

Mild steel; Pyrrolidines; Polarization; EIS; Acid corrosion

ABSTRACT

A series of pyrrolidines were synthesised using phenylalanine and isatin with dipolarophiles. Azomethine ylide was first formed from phenylalanine and isatin which undergoes 1,3-dipolar cycloaddition with dipolarophiles yielding pyrrolidines. The corrosion inhibition property of the pyrrolidines towards mild steel corrosion in 1M HCl has been investigated using electrochemical techniques. The results showed that the presence of pyrrolidines in the acid solution suppress the corrosion rate of mild steel. The inhibition efficiency was found to dependent on the substitutions in the ring and with the concentration of the particular species in acid solution. Potentiodynamic polarizations studies revealed the mixed type inhibition nature of the compounds. Electrochemical impedance spectroscopy suggested the charge transfer controlled mechanism of corrosion inhibition.

INTRODUCTION

Mild steel is an important material of choice due to low cost and easy availability. Hydrochloric acid is one of the most important pickling acids, which is widely used in steel and ferrous alloy industry for acid cleaning, acid descaling, oil well oxidizing and other petrochemical processes. The main problem in using mild steel in acidic solution is its degradation due to corrosion. Prevention of corrosion of mild steel in acidic solutions is of practical importance. A possible solution to protect mild steel in acidic environment is the application of corrosion inhibitors or coatings.

Previous studies had revealed the relationship between structural characteristics of the organic compounds and their inhibiting effects, Emregul and Atakol, 2003; Khaled, 2003. It had been suggested that the most effective factors for the inhibiting effects are: the lone pair electrons of the electronegative atoms (such as, N, S, P, O, etc.) Zhao *et al* (2005), the pi-electrons of the unsaturated bonds, Emregul and Atakol, 2003 (such as, double bonds or triple bonds, etc.) and the delocalized pi-electrons of the plane conjugated systems including all kinds of aromatic cycles, Khaled, 2003. When a metal surface is dipped in an electrolytic solution the sorption and transport of ions in the presence of water and oxygen would influence the corrosion behavior of metal.

In many cases, the inhibition is related to the adsorption of the inhibitor on the metal surface forming a barrier layer which separates the metal from the corrosive media. According to the type of inhibitor species and the nature of metal, Abdennabi *et al* (1998) adsorption may be chemical or physical adsorption, (Quraishi and Sardar, 2002; Stankovic and Vukovic, 1996).

Heterocyclic compounds such as pyrrolidines, pyrrolines, and pyrroles are reported as good corrosion inhibitors, Quraishi and Khan, 2005; Kadhum *et al* (2014). In the present work, spiropyrrolidines were synthesized from a heterocyclic compound, Isatin and its inhibition effect on the corrosion behavior of mild steel in 1M HCl was studied using potentiodynamic polarization and electrochemical impedance spectroscopy method. The adsorption process was tested using Langmuir and Temkin adsorption isotherms.

Experimental details

General procedure for synthesis of Spiropyrrolidines Bearing Oxindole (I a-d)

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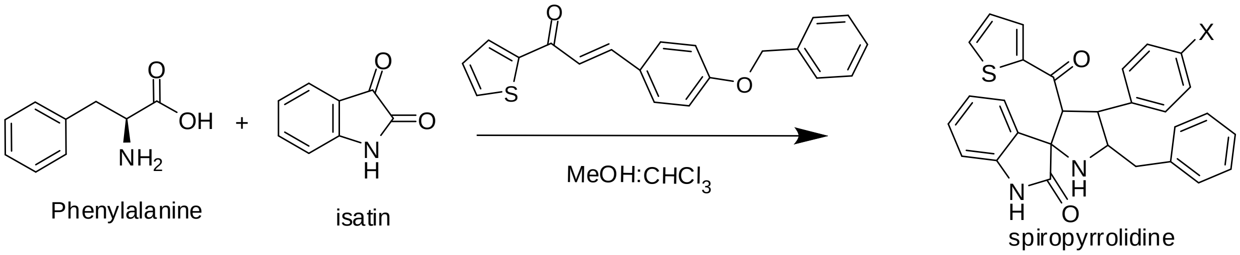
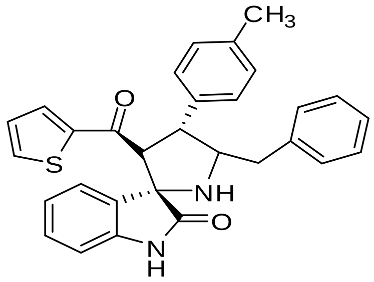
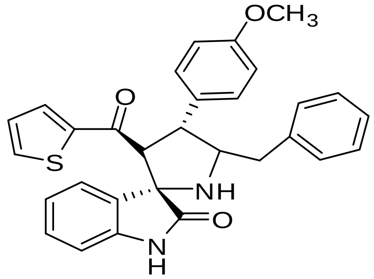
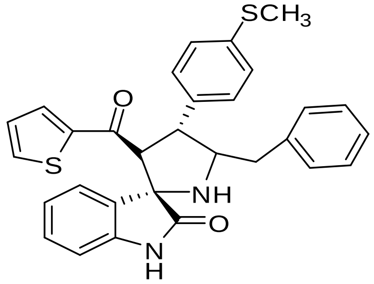
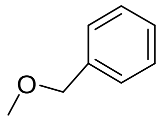
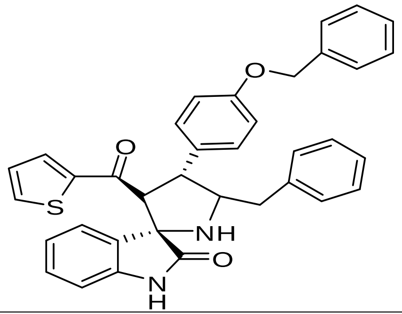
Initially the reaction proceeds through decarboxylative condensation between isatin and Phenylalanine to generate the azomethine ylide which subsequently undergoes 1,3-dipolar cycloaddition with the (E)-3-aryl-1-(thiophen-2-yl) prop-2-en-1-ones to afford spiropyrrolidine derivatives with oxindole moieties, **Scheme 1**.

The regioselective outcome of the spiro derivatives I a-e was confirmed by using IR, 1H NMR and 13C NMR spectroscopic and by elemental analysis data, Geethanjali and Thangamani, 2014. The structures of the synthesized compounds are given in **Table 1**. The compounds were dissolved in ethanol and diluted with double distilled water to get stock solution.



Scheme 1 Reaction between Phenylalanine and Isatin

Table 1 Details of the studied inhibitors

group 'X'	Name	Structure	Abbreviation
$-CH_3$	5'-benzyl3'-(thiophene-2-carbonyl)-4'-(4-methylphenyl)-spiro[indoline-3,2'-pyrrolidin]-2-one		MPIP Ia
$-OCH_3$	5'-benzyl3'-(thiophene-2-carbonyl)-4'-(4-methoxyphenyl)-spiro[indoline-3,2'pyrrolidin]-2-one		MOPIP Ib
$-SCH_3$	5'-benzyl-3'-(thiophene-2-carbonyl)-4'-(4-methylthiophenyl)-spiro[indoline-3,2'- pyrrolidin]-2-one		MTPIP Ic
	5'-benzyl-3'-(thiophene-2-carbonyl)-4'-(4-(benzyloxy)phenyl)-spiro[indoline-3,2'-pyrrolidin]-2-one		BOPIP Id

Electrochemical Measurements

The experiments were performed with mild steel specimens having composition (% by weight) 0.196 Mn, 0.106 C, 0.027 P, 0.022 Cr, 0.016 S, 0.012 Ni, 0.006 Si, 0.003 Mo and remainder Fe. Electrochemical measurements were performed using IVIUM Compactstat potentiostat/galvanostat. Polarization experiments were carried out in a conventional three electrode cell with a platinum foil as counter electrode, saturated calomel electrode (SCE) as reference electrode and mild steel specimens having an exposed area of 1 cm^2 as working electrode. The working electrode was held in the test environment for half an hour prior to each experiment to attain stable open circuit potential (OCP) value. The anodic and cathodic polarization curves were recorded at a constant sweep rate of 1 mV/s from -100 mV to +100 mV. The corrosion currents for different sets of solution were found from the extrapolation of the polarization curves back to the OCP. The inhibition efficiency was calculated using the following equation,

$$IE_{Icorr}\ \% = \frac{I^{o}_{corr} - I^{i}_{corr}}{I^{o}_{corr}} * 100 \qquad (1)$$

where I^{o}_{corr} and I^{i}_{corr} represents the uninhibited and inhibited corrosion current densities.

Impedance spectra were obtained in the frequency ranges of 10 kHz to 0.1 Hz with an alternating current of 10 mV amplitude, at the corrosion potential. All tests have been performed at 30 ^{o}C in open aerated solutions under static conditions. Inhibition efficiency was calculated from the charge transfer resistance values as,

$$IE_{Rct}\ \% = \frac{R^{i}_{ct} - R^{o}_{ct}}{R^{i}_{ct}} * 100 \qquad (2)$$

where R^{o}_{ct} and R^{i}_{ct} represents the uninhibited and inhibited charge transfer resistance. The surface coverage values, θ were calculated as θ=IE/100 and used for testing adsorption isotherms.

RESULTS AND DISCUSSIONS

Characterization of the compounds

5'-benzyl3'-(thiophene-2-carbonyl)-4'-(4-methylphenyl)-spiro[indoline-3,2'-pyrrolidin]-2-one (MPIP): Yield 90%, Melting point 188-192˚C, IR (KBr, cm^{-1}) 1712 (thiophenyl carbonyl), 1633 (oxindole carbonyl). ^{1}H NMR (400MHz, $CDCl_3$) δ: 1.65 (-NH of pyrrolidine ring) (s,1H), 2.69 (H5-CH_2-) (dd, 1H, J = 7.8 & 13.8 Hz); 2.99 (H5-CH_2-) (dd, 1H, J = 2.4 &12.8 Hz), 3.85 (H4) (t, 1H, J = 10.8 Hz); 4.40 (H3) (d, 1H, J = 10.8 Hz); 4.20-4.25 (H5) (m, 1H); 6.50 (1H, d, J = 8 Hz); 6.80 (t, 1H, J = 4 .4Hz); 6.88-6.91 (m, 2H); 6.98-7.14 (m, 1H); 7.16 (d, 2H J = 8.4 Hz); 7.19-7.21(m, 3H); 7.24-7.27 (m, 2H); 7.42 (d, 2H, J = 8 Hz); 7.78 (-NH of oxindole ring) (s,1H).

5'-benzyl3'-(thiophene-2-carbonyl)-4'-(4-methoxyphenyl)-spiro[indoline-3,2'pyrrolidin]-2-one (Ib): Yield 92%, Melting point 198-201˚C,IR(KBr, cm-1) 1712 (thiophenyl carbonyl), 1639 (oxindole carbonyl).1H NMR (400MHz, CDCl3) δ: 1.26,(s,1H), 2.68 (H5-CH2) (dd, 1H, J = 7.6&14 Hz), 2.99 (H5-CH2) (dd, 1H, J = 3.2 &14 Hz), 3.78 (O-CH3) , (s,3H),3.83 (H4) (t,1H,J=10.4Hz), 4.17-4.23 (H5)(m,1H) 4.37 (H3) (d, 1H,J=10.8Hz), 6.51(1H,d, J = 8 Hz), 6.81 (t, 1H, J = 4.2Hz), 6.87-6.90(m ,4H) 6.99-7.02(m,1H),) 7.16-7.20(m,4H),7.23-7.27(m,2H)7.33(d ,3H J=4.4Hz), 7.45(d ,3H, J=8.8), 7.78,-NH(s,1H).

5'-benzyl-3'-(thiophene-2-carbonyl)-4'-(4-methylthiophenyl)-spiro[indoline-3,2'- pyrrolidin]-2-one (Ic): Yield 91%, Melting point 199-202˚C, IR(KBr, cm-1) 1712 (thiophenyl carbonyl), 1633(oxindole carbonyl).1H NMR (400MHz, CDCl3) δ: 1.65, -NH of pyrrolidine ring (s,1H),), 2.46(-SCH3)- (s,1H) 2.69 (H5-CH2) (dd, 1H, J = 7.6&14 Hz), 2.98 (H5-CH2) (dd, 1H, J = 3.2 &14 Hz), 3.83 (H4) (t,1H,J=10.6Hz), 4.20-4.25 (H5)(m,1H) 4.37 (H3) (d, 1H, J=10.8Hz),6.51(d,1H J=8Hz), 6.78-6.99 (m,1H), 6.86-6.90(m,1H),6.96,(M,1H J=8.8Hz), 6.97-7.0 (m,1H),7.15-7.20 (m,3H)7.22-7.28 (m,2H),7.30-7.33 (m,3H),7.32-7.34 (m,2H) 7.38-7.42(m,2H), 7.45 (d,1H, J=8.8Hz), 7.46 ,(-NHof oxindole ring,),(s,1H).

5'-benzyl-3'-(thiophene-2-carbonyl)-4'-(4-(benzyloxy)phenyl)-spiro[indoline-3,2'-pyrrolidin]-2-one (Id): Yield 85%, Melting point 190-194˚C,IR(KBr, cm-1) 1712 (thiophenyl carbonyl), 1608 (oxindole carbonyl).1H NMR (400MHz, CDCl3) δ:2.69 (H5-CH2) (dd, 1H, J = 7.6&14 Hz), 2.99 (H5-CH2) (dd, 1H, J = 3.2 &14 Hz), 3.83 (H4) (t,1H,J=10.6Hz), 4.18-4.23 (H5) (m,1H) 4.37 (H3) (d, 1H,J=10.8Hz), 5.02 -O-CH2-(s,2H),), 6.51(d,1H J=8Hz), 6.78-6.99 (m,1H), 6.86-6.90 (m,1H),6.96,(m,1H J=8.8Hz), 6.97-7.0 (m,1H), 7.15-7.20 (m,3H), 7.22-7.28 (m,2H), 7.30-7.33(m,3H), 7.32-7.34 (m,2H) 7.38-7.42 (m,2H), 7.45 (d,1H, J=8.8Hz), 8.1(-NH),(s,1H).

Electrochemical measurements

Potentiodynamic Polarization Studies

Figure 1 represents the potentiodynamic polarization plots for mild steel in 1 M HCl in the absence and presence of compounds MPIP, MOPIP, TMPIP and BOPIP respectively. The Tafel extrapolation method was used to derive the electrochemical parameters which are provided in Table 2. The presence of pyrrolidine derivatives shifted the cathodic and anodic curves towards positive potential region. Both cathodic and anodic Tafel slopes were changed with increase in the inhibitor concentration. This clearly indicates that the inhibitor controlled both the cathodic and anodic reactions and thus behaved as a mixed type inhibitors, Abdel-Rehim *et al* (2006). It is also observed that the E_{corr} values did not change significantly in the presence of the inhibitors suggesting that the inhibitors are of mixed type inhibitors.

The corrosion current densities, I_{corr} of the plots were found to be dependent on the inhibitor concentration. The I_{corr} value for mild steel specimen in 1M HCl is 0.3469 mA/cm^2 and it decreases as the concentration of the pyrrolidine derivatives increases. The I_{corr} value for the 1 mM concentration of MPIP is 0.0504; MOPIP is 0.0658; MTPIP is 0.0001 and TPCIP is 0.0718 mA/cm^2. This indicates that out of the four inhibitors used MTPIP showed maximum suppression of the corrosion current density. The inhibition efficiencies calculated according to equation 1 is also provided in Table 2.

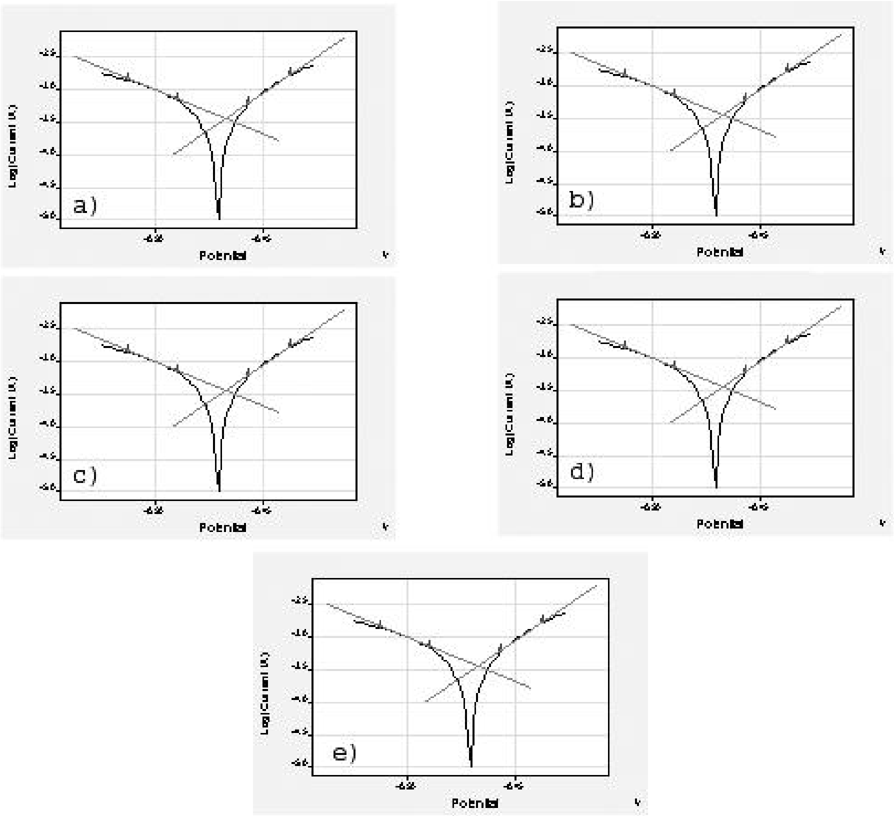


Figure 1 Potentiodynamic polarization plots for mild steel in a) 1 M HCl b) 1 mM MPIP c) 1 mM MOPIP d) 1 mM MTPIP and e) BOPIP in 1 M HCl

Table 2 Polarization parameters for mild steel in the presence and absence of inhibitors

Conc.	ba mV/dec	bc mV/dec	E_{corr} mV	R_p (Ohm)	CR (mm/y)	I_{corr} (mA/cm^2)	IE $_{Icorr}$ (%)
1 M HCl	44	75	-467.6	34.78	1.1350	0.3469	-
MPIP							
0.3 mM	65	90	-496.8	275.8	0.2316	0.0708	79.59
0.5 mM	37	62	-502.9	295.5	0.1648	0.0504	85.48
1 mM	48	81	-510.4	330.5	0.1515	0.0463	86.65
MOPIP							
0.3 mM	42	98	-501.1	100.9	0.4386	0.1341	61.34
0.5 mM	33	93	-508.6	218.0	0.2450	0.0746	78.41
1 mM	56	108	-504.2	311.3	0.2153	0.0658	81.03
MTPIP							
0.3 mM	69	135	-482.8	203.2	0.4068	0.1243	64.16
0.5 mM	66	133	-479.3	269.7	0.2953	0.0009	99.73
1 mM	65	135	-470.0	289.6	0.2736	0.0001	99.97
BOPIP							
0.3 mM	35	95	-510.4	187.7	0.2559	0.0782	77.45
0.5 mM	65	114	-512.5	292.8	0.2488	0.0760	78.08
1 mM	67	104	-517.4	314.9	0.2348	0.0718	79.32

The inhibition efficiency increases as the concentration of the compounds increased from 0.3 to 0.5 and 1 mM. As the concentration of the compounds increase the number of molecules available for adsorption on the metal surface increases. This leads to increase in surface coverage values and hence higher inhibition efficiency. Out of the four compounds, MTPIP showed higher inhibition efficiency of 99.97% followed by MPIP (86.65 %), MOPIP (81%) and BOPIP (79.3%). The higher inhibition efficiency of MTPIP is due to the presence of ‘S-CH_3’ it favors the adsorption on the mild steel surface. In MPIP the adsorption of the compound is more favorable compared to MOPIP. In BOPIP the benzyl oxy group disfavors the adsorption process. The steric hindrance plays an important role in BOPIP which lowers the inhibition efficiency to 79 %.

AC Impedance Studies

Nyquist representations of mild steel in uninhibited and inhibited solutions containing 1 mM concentration of the tested inhibitors are given in Figure 2. It is clear from the representations that the plots are not perfect semicircles, but are depressed one. This is due to the frequency dispersion occurred during the formation of double layer at the metal/solution interface, Mansfeld *et al* (1982). Various parameters derived by fitting the impedance data for an equivalent circuit consist of R_{ct}, C_{dl} are summarized in Table 2. The R_{ct} values (Table 3) increased as the concentration of inhibitors increased, whereas C_{dl} values (Table 3) decreased. This is attributed to the increase in the surface coverage by the inhibitors leading to an increase in inhibition efficiency. A decrease in the local dielectric constant and/or an increase in the thickness of the electrical double layer are responsible for the decrease in C_{dl} values. The change in R_{ct} and C_{dl} values were caused by the gradual replacement of water molecules by the adsorption of the pyrrolidines on the metal surface, decreasing the extent of metal dissolution, Hackermann *et al* (1966).

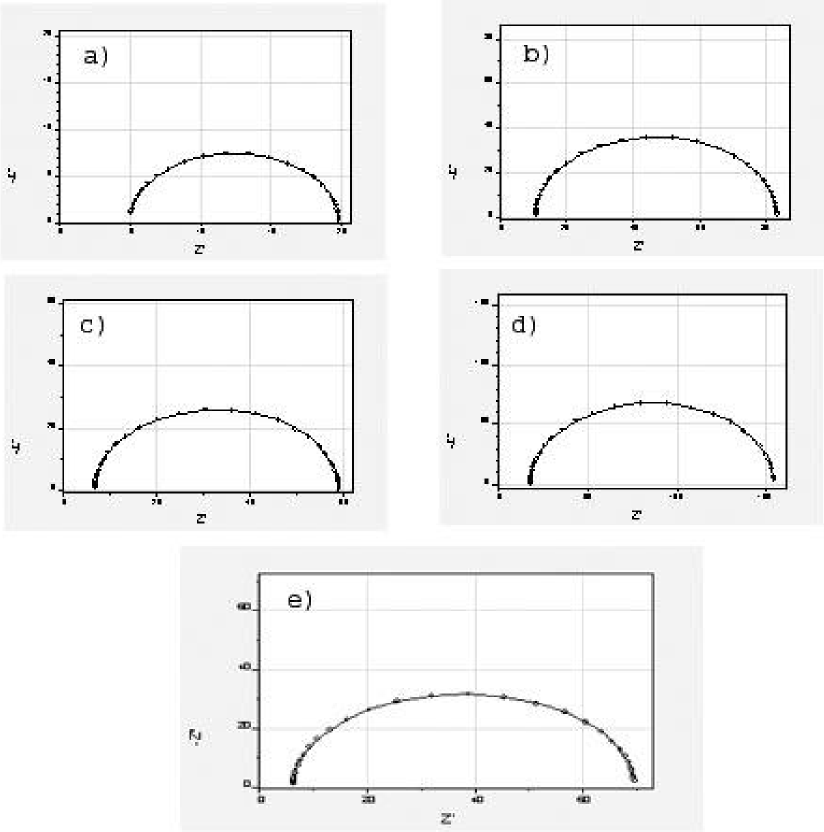


Figure 2. Nyqist representations for mild steel corrosion in the absence and presence of MPIP, MOPIP, MTPIP, BOPIP

Conc.	Rs Ohms	R_{ct} Ohms	C_{dl} x 10^{-5} F	Error 10^{-9}	–	IE_{Rct} (%)
1 M HCl	5.00	14.86	1.55	5.22	-	-
MPIP						
0.3 mM	8.91	63.00	1.57	3.69	0.7641	76.41
0.5 mM	9.67	63.53	1.68	3.97	0.7661	76.61
1 mM	10.62	72.38	1.78	4.27	0.7947	79.47
MOPIP						
0.3 mM	6.26	35.30	1.30	3.52	0.5794	57.94
0.5 mM	5.61	37.81	1.76	4.12	0.6067	60.67
1 mM	6.79	52.34	1.32	3.09	0.7159	71.59
MTPIP						
0.3 mM	1.50	97.6	5.73	12.1	0.8477	84.77
0.5 mM	1.52	116.00	5.24	10.7	0.8723	87.23
1 mM	1.82	136.02	5.43	10.8	0.8907	89.07
BOPIP						
0.3 mM	6.67	55.00	1.66	3.48	0.7300	73.00
0.5 mM	9.05	63.30	1.45	3.24	0.7651	76.51
1 mM	6.30	78.10	1.48	3.67	0.8096	80.96

Adsorption Isotherms

The adsorption isotherms can give valuable information about the metal inhibitor interactions. The inhibitor efficiency depends on the type and number of active sites at the metal surface, the charge density, the molecular size of the inhibitor and the metal-inhibitor interactions. The degree of surface coverage (θ) obtained for the four studied inhibitors at room temperature has been tested graphically for fitting a suitable isotherm. The plots of logC Vs log(C/θ) yield a straight line (Lagrenee *et al* 2002), proving that the adsorption of the composites on the mild steel surface obeys the Langmuir adsorption isotherm. Langmuir isotherms for the pyrrolidine derivatives are provided in Figure for the results of impedance measurements.

The correlation coefficients for the isotherms are presented in the plots for the studied inhibitors. The correlation coefficients are found to be more close to unity proving the fitness of the data with the Langumir isotherm.

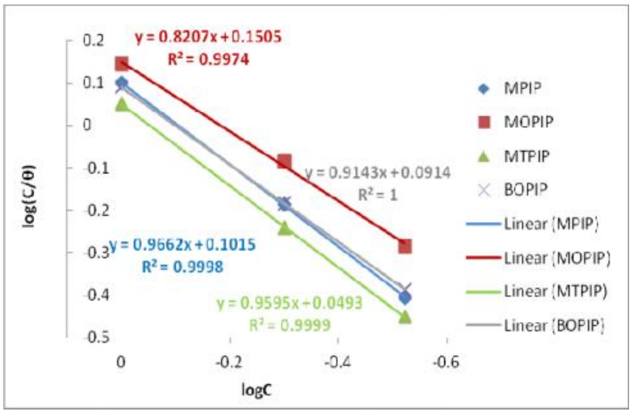


Figure 3 Langmuir Adsorption Isotherms – Impedance measurements

Thermodynamic Parameters for Adsorption

The free energy of adsorption, (ΔG_{ads}) of the inhibitors can be calculated by using the following equation (Bentoss *et al* 2005), where, C is the concentration the inhibitor.

$$\log C = [\log \theta / (1- \theta)] + 1.74 + (\Delta G/ 2.303\ RT) \qquad (3)$$

The calculated values of ΔG_{ads} at different concentrations of the pyrrolidines are listed in table 3. The negative values of ΔG_{ads} suggest that the adsorption of the pyrrolidines derivatives is a spontaneous process. And also the values are less than 40 kJ/mol. These lower values are attributed to the electrostatic interaction between the charged composites and the charged metal surface (physisorption).

Mechanism of inhibition

The process of adsorption of organic inhibitors are influenced by the nature and charge of the metal surface, the chemical structure of the inhibitor, nature of the aggressive medium and the interaction between the inhibitor molecules and the metallic surface, (Oguzie *et al* 2004). Water molecules first adsorbed on the surface of mild steel immersed in the aqueous medium, (Cafferty and Hackermann, 1972). The chloride ions from the medium gradually replace the water molecules and get adsorbed on the mild steel surface. The heterocyclic nitrogen containing compounds are relatively ineffective in corrosion mitigation of iron and steel in H_2SO_4 solutions unless certain anions especially halides and pseudo halide ions are present, (Abdel Rahim *et al* 1997). This evidencing the fact that chloride ions of the medium first get adsorbed on the positively charged metal surface which in turn facilitates the adsorption of protonated species. Moreover, there are more numbers of electron rich centers in the studied pyrrolidines which facilitates the high extent of adsorption. Thus the compounds may block the micro-anodes and/or micro-cathodes that are generated on the metal surface when in contact with the aggressive medium. This retarded the subsequent dissolution of the metal thereby increasing the inhibition efficiency, Jayalakshmi and Muralidharan, 1998.

CONCLUSION

From the above studies, it can be concluded that, the pyrrolidines synthesized from phenylalanine and isatin with dipolarophiles, results in more than 80% inhibition efficiency. Highest inhibition efficiency was obtained for MTPIP followed by MPIP, MOPIP and BOPIP. The inhibition efficiency increased with the concentration of pyrrolidines. For the studied inhibitors the polarization and electrochemical impedance measurements showed the same trends. The cathodic and anodic Tafel slopes reveal that the synthesized inhibitors are mixed type inhibitors. The closest description of the adsorption behavior of the composites is given by Langmuir adsorption isotherms. The values of free energy of adsorption indicate the spontaneity and physical nature adsorption of pyrrolidines on the mild steel surface.

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How to cite this article:

Geethanjali K and Ali Fathima Sabirneeza A.2016, Spiropyrrolidine Derivatives as Corrosion Inhibitor for mild Steel in 1m HCL. *Int J Recent Sci Res.* 7(4), pp. 10189-10195.

ISSN 0976-3031

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