CORROSION BEHAVIOUR OF MILD STEEL IN ACID SOLUTIONS WITH

S-AITO

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Abstract

S-Acetyl Isothiourea Oxalate (S-AITO) was synthesized in the laboratory and this influence on the inhibition of corrosion of mild steel in 1.11 N hydrochloric and 1.12 N sulphuric acids was investigated by weight loss and potentiostatic polarization techniques at 303K, 353K and 403K. These results were confirmed by the impedance technique. The inhibition efficiency increased with increase in concentration of inhibitor and decreased with rise in temperature from 303K to 403K. The maximum inhibition efficiency of S-AITO was found to be 98.42% (0.5% of S-AITO) at 303K in sulphuric acid. The adsorption of this compound on the mild steel surface from the acids has been found to obey Temkin's adsorption isotherm. The potentiostatic polarization results revealed that S-AITO was a mixed type inhibitor. Some thermodynamic parameters i.e., activation energy (Ea), free energy of adsorption (ΔG_{ads}), enthalpy of adsorption (ΔH) and entropy of adsorption (ΔS) were also calculated from weight loss data.

Keywords

Mild steel, hydrochloric acid, sulphuric acid, Temkin's adsorption isotherm, potentiostatic polarization, impedance.

Introduction

Concentrated mineral acids are used extensively in pickling, cleaning, descaling and oil well acidising of metallic materials cause damage of corrosion [1,2]. It has been

speculated that organic inhibitors are more effective with iron and that polar organic compounds containing sulphur and nitrogen are good corrosion inhibitors for the acidic solutions of metals [3, 4].

Many organic inhibitors with hetero atoms have been studied [5-10]. High electron density of the sulphur and nitrogen atoms in these heteroatoms, help the organic molecules to get chemisorbed onto the metal surface [8]. Due to the aggressiveness of hydrochloric acid and sulphuric acid solutions against structural materials, such as carbon steel, the use of corrosion inhibitors is usually required to minimize the corrosion attack [11-14].

Therefore, in this investigation, the corrosion of mild steel in 1.11N HCl and 1.12N H_2SO_4 solutions in the absence and presence of S-AITO at 303K to 403K has been studied by weight loss, potentiostatic polarization and impedance techniques. It is aimed at attempting to predict the thermodynamic feasibility in inhibition via, surface coverage of the mild steel by adsorbed S-AITO. The adsorption characteristic of S-AITO was studied in order to across the adsorption isotherm that the experiment data can fit and some thermodynamic parameters of adsorption calculated.
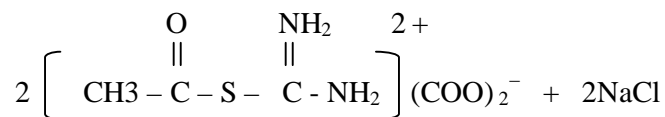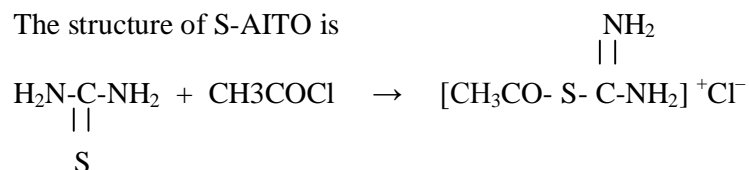
Experimental

Weight loss measurements

Mild steel specimens were cut to the size of 5cm x 1cm from the mild steel sheets having the following percentage composition Fe=99.686, Ni=0.013, Mo = 0.015, Cr = 0.043, S= 0.014, P=0.009, Si-0.007, Mn = 0.196, C=0.017. Weight loss measurements were performed as per ASTM method described previously [15-17]. Weight loss measurements were carried out in 1.11N HCl and 1.12N H_2SO_4 acids with inhibitor S-Acetyl Isothiourea Oxalate (S-AITO) in the concentration range of 0.1% to 0.5% at 303K to 403K for an immersion period of 2 hours with and without inhibitor. All the solutions were prepared using AR grade chemicals with double distilled water.

Inhibitor was synthesized in the laboratory. The reaction between acetyl chloride and thiourea gives compound known as S-acetylthiuronium chloride or S-acetyl isothiourea hydrochloride. On mixing a solution of the alkali salt of carboxylic acid (sodium oxalate), the S-acetyl isothiourea oxalate is formed rapidly. Synthesized compound was characterized through the spectral data and the purity was confirmed by thin layer chromatography (TLC).

The structure of S-AITO is

$$H_2N-C-NH_2 \ + \ CH3COCl \ \rightarrow \ [CH_3CO- S- \overset{\overset{NH_2}{||}}{C}-NH_2] \ ^+Cl^-$$
$$\underset{S}{\overset{||}{|}}$$

$$2[\ CH_3CO - S - \overset{\overset{NH_2}{||}}{C} - NH_2]^+Cl^- \ + \ (COONa)_2 \ \rightarrow$$

$$2 \left[\ CH3 - \overset{\overset{O}{||}}{C} - S - \ \overset{\overset{NH_2}{||}}{C} - NH_2 \ \right]^{2+} (COO)_2^- \ + \ 2NaCl$$

Potentiostatic polarization /A.C. impedance measurements

For potentiostatic measurements, mild steel electrode of 1 cm x 1cm dimensions with stem was cut. The stem and the face of the electrode were marked with araldite, so as to expose an area of 1 cm^2. The electrodes are polished using 1/0, 2/0, and 3/0and 4/0 emery papers and degreased with trichloroethylene. Potentiostatic polarization measurements were carried out in three-electrode cell using BAS-100A model instrument. The potential of the test electrode was measured with respect to SCE and platinum was used as auxillary electrode and the experiments were carried out at 302K. The polarization measurements were carried out from a potential range of -200mV to +200mV with respect to open circuit potential, at a scan rate of 1mV/sec. Linear polarization experiments were carried out by applying the potential of ± 20 mV from the corrosion potential and resultant current was measured, then E versus i were made in a linear scale to get linear polarization plots and the slope of the plots

have the polarization resistance R_p. In Impedance measurements, Solartron Electrochemical measurement unit (1280B) was used. Impedance measurements were carried out at corrosion potential. The A.C. amplitude of 10mV was applied and the frequency was varied from 10 KHz to 10 MHz.

Results and Discussion

Weight loss measurements

Table I shows the values of inhibition efficiency (IE %), surface coverage (θ) and corrosion rate obtained for different concentrations of inhibitor (S-ATAO) in 1.11N HCl and 1.12N H$_2$SO$_4$ acids for immersion period of 2 hours at 303K to 403K. From the weight loss value, the inhibition efficiency (IE %) and surface coverage were calculated using the following equation [18, 19].

Inhibition efficiency (IE %) $= \dfrac{Wu - Wi}{Wu} x100 - - - - - 1$

Surface Coverage (θ) $= \dfrac{Wu - Wi}{Wu} - - - - - 2$

Where Wu and Wi are the corrosion rates for mild steel in the absence and presence of inhibitor (S-AITO) respectively at the same temperature.

It clearly indicates that the addition of inhibitor to the acids (1.11N HCl and 1.12N H$_2$SO$_4$) has reduced the corrosion rate. The inhibition efficiency increased with increase in concentration of inhibitor and decreased with rise in temperature from 303K to 403K. Fig. 1 and 2 show the relation between the inhibition efficiency and temperature for 0.1% to 0.5% of S-AITO in mild steel in 1.11N HCl and 1.12N H$_2$SO$_4$ respectively. The order of the efficiency is decreased with increasing temperature. These results indicate that the process film start to break down with increasing temperature. The values of corrosion rate and inhibition efficiency of the inhibitor were found to depend on the molecular structure of

inhibitor. The maximum inhibition efficiency of S-AITO was 79.21% in 1.11N hydrochloric acid and 98.42% in 1.12N sulphuric acid at 0.5% (S-AITO). The maximum inhibition efficiency was obtained in sulphuric acid but not in hydrochloric acid. This is due to that HCl contains chloride ions; generally chloride ions stimulate the corrosion rate.

Thermodynamic consideration

Table 2 shows the calculated values of activation energy Ea(KJ/mole), free energy of adsorption ΔG_{ads}(KJ/mole), enthalpy of adsorption ΔH (KJ /mole) and entropy of adsorption ΔS (KJ mole^{-1} K^{-1}) for mild steel corrosion in 1.11 N HCl and 1.12 N H$_2$SO$_4$ with and without inhibitor. Energy of activation (Ea) has been calculated from Arrhenius equation [20-22].

$$\log \frac{p_2}{p_1} = \frac{Ea}{2.303 \times R} \left[\frac{1}{T_1} - \frac{1}{T_2} \right] \text{--------------------3}$$

Where P$_1$ and P$_2$ are the corrosion rate at temperature T$_1$ and T$_2$ respectively. Ea values given in Table 2 shows that the Ea values for the corrosion of mild steel in 1.11N HCl and 1.12N H$_2$SO$_4$ are 39.37 KJ /mole and 20.90 KJ /mole respectively. In acid containing inhibitor, the Ea values are found to be higher than that of the uninhibited system. The higher values of Ea indicate physical adsorption of the inhibitor on metal surface [23]. It is clear that the activation energy increases regularly with increasing the efficiency of the inhibitor.

The Free energy of adsorption (ΔG_{ads}) at different temperature was calculated from the following equation [24].

$$\Delta G_{ads} = -RT \ln (55.5K) \qquad \text{----------------------4}$$

and K is given by

$$K = \frac{\theta}{C (1- \theta)} \text{--------------------5}$$

Where θ if surface coverage, C is concentration of inhibitor in mole / lit and K is equilibrium constant. From Table 2, the negative values of ΔG_{ads} obtained indicate the spontaneous adsorption of the indicator and are usually characteristics of strong interaction with the metal surface. It is found that the ΔG_{ads} are less than (less -ive values) – 40 KJ/ mole indicate that the inhibitor is physically adsorbed on the metal surface [25-26].

The enthalpy of adsorption ΔH and entropy of adsorption ΔS can be calculated form the following equations

$$\Delta H = Ea - RT \quad \text{-----------------------------6}$$

$$\Delta G = \Delta H - T \Delta S \quad \text{-------------------------7}$$

ΔS can be easily calculated at 303K to 403K for the different concentration of S-AITO. It is also observed that ΔS is increased with increasing the efficiencies of the inhibitor. This is opposite to that we expect, since the adsorption is an exothermic process and is always accompanied by a decrease of entropy. Ateya et al [27] have described this situation as the adsorption of the organic compound, which is accompanied by desorption of water molecules of the surface. Thus while the adsorption process is believed to be exothermic and associated with a decrease in entropy of the solute the opposite is true for the solvent. Therefore, this gain in entropy that accompanied the substitution adsorption process is attributed to the increase in solvent entropy.

Adsorption isotherms

The electrochemical process on the metal surface is likely to be closely related to the adsorption of inhibitors [28] and the adsorption is known to depend on the chemical structure of the inhibitors [29-30]. The adsorption of the inhibitor molecules from aqueous solution can be regarded as quasisubsitution process [29] between the organic compounds in the aqueous phase, Org $_{(aq)}$ organic aqueous and water molecules at the electrode surface, $H_2O_{(S)}$

$$Org_{(aq)} + x \; H2O_{(s)} \quad = \quad Org_{(s)} \; + x \; (H_2O)_{(aq)} \; \text{-----------} 8$$

Where x is the size ratio, is the number of water molecules displaced by one molecule of organic inhibitors.

Adsorption isotherms are very important determining the mechanism of organo – electro chemical reactions. The most frequently used isotherms are those of Langmuir, Frumkin, Parons, Temkin, Flory – Huggins and Bockris Sinkles [32-35]. All these isotherms are of the general form.

$$f\,(\theta, x)\, exp\,(-a\,\theta) \quad = KC \text{------------- } 10$$

Where f (θ, x) is the configurational factor that depends essentially on the physical model and assumptions underlying the derivation of the isotherm [36]. The mechanism of inhibition of corrosion is generally believed to be due to the formation and maintenance of a protective film on the metal surface.

The plot of surface coverage (θ) obtained by weight loss method versus log C (concentration) for different concentrations of the compound show a straight line indicating that the adsorption of the compounds from acids on mild steel surface follow Temkin's adsorption isotherm. This also pointed to result of their adsorption on the metal surface. Fig 3 and 4 shows the Temkin's adsorption isotherm for S-AITO.

Potentiostatic polarization measurements

The polarization behaviour of mild steel functioning as cathode as well as anode in the test solutions is shown in Fig 5 and 6 for 1.11N HCl and 1.12N H_2SO_4 at 303K for S-AITO. Similar curves were also obtained for 1.11N HCl and 1.12N H_2SO_4 at 353K and 403K and the electrochemical data obtained from the studies are shown in Table 3. It is evident that S-AITO bring about considerable polarization of the cathode as well as anode. It

was, therefore, inferred that the inhibitive action is of mixed type. The cathodic and anodic Tafel slopes increased with increasing inhibitor concentrations and the increase was predominant in the case of the former indicating that the cathodic inhibition was dominating through the inhibitive active is of mixed nature. The non-constancy of Tafel slopes for different inhibitor concentration reveals that the inhibitor act through their interference in the mechanism of the corrosion processes at the cathode as well as the anode.

The corrosion parameters deduced from Tafel polarization such as corrosion current i_{corr}, corrosion potential E_{corr}, Tafel constant ba and –bc, inhibition efficiency and Rp are given in Table 3. The i_{corr} values were decrease with the increasing concentration of inhibitor. The inhibition efficiencies were determined from the values of corrosion current and the inhibition efficiency values were found to show same trend, with those obtained from mass loss measurements.

Impedance measurements

A.C. impedance measurements were carried out at room temperature for corrosion of mild steel in 1.11N HCl and 1.12N H_2SO_4 after immersion for about 10 minutes. The Nyquist plots for mild steel in uninhibited acid and for the various concentrations of inhibitor are shown in Fig 7 and 8. The impedance parameters and the IE% are given in Table 4.

The charge transfer resistance (R_{ct}) value for mild steel in uninhibited HCl and H_2SO_4 significantly changes after the addition of inhibitor. The R_{ct} values increased with increase in inhibitor concentration. The fact is advocated by the increase in inhibitor efficiency. The semicircular nature of Nyquist plots obtained for all experiments indicates that the corrosion of mild steel is controlled by charge transfer process [37]. The double layer capacitance (C_{dl}) decreased with increasing inhibitor concentration. The decrease in C_{dl} values in presence of inhibitor indicates the fact that these additives inhibit corrosion by adsorption on the metal surface [37].

The inhibition efficiency obtained from A.C. impedance measurements are not in very good agreement with these obtained from weight loss studies. Some differences are always noticed when corrosion rates and inhibition efficiencies determined by conventional weight loss method are compared with those obtained from electrochemical techniques. The difference observed can be attributed due to the fact weight loss methods give average corrosion rates where as electrochemical methods give instantaneous corrosion rates [38], weight loss measurements are experiments of long duration which helps in the formulation of inherent and continuous film on the metal surface. This leads to the complete shielding of the metal surface from the corrosive experiments. But electrochemical studies are experiments of short duration and the time will not be enough for the formation of the thick continuous film. This leads to slight difference in the inhibition efficiency [39,40].

Conclusions

The conclusions reported in this paper are the following

1. S-AITO acted as efficient corrosion inhibitor in 1.11N HCl and 1.12N H_2SO_4.

2. The inhibition efficiency increased with increase in concentration of inhibitor (0.1to 0.5%) and decreased with increase in temperature from 303K to 403K.

3. The maximum inhibition efficiency of S-AITO was 79.12% in 1.11N HCl and 98.42% in 1.12N H_2SO_4 (0.5% of S-AITO).

4. The thermodynamics values obtained from this study Ea, ΔH, and ΔS, indicated that the presence of the inhibitor increase activation energy and the negative values of ΔG_{ads} indicate spontaneous adsorption of the inhibitor on the surface of the mild steel.

5. S-AITO inhibitor corrosion by adsorption mechanism and the adsorption of this compound from acid solution follows Temkin's adsorption isotherm at all the concentrations.

6. From the potentiostatic polarization studies, S-AITO acted as mixed type inhibitor.

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Table I

Calculated values of corrosion rate, inhibition efficiency and surface coverage for S-AITO in 1.11N HCl and 1.12 N H_2SO_4 from weight loss method

Temp.	Conc.of S-AITO (%)	1.11N HCl			1.12N H_2SO_4		
		Corrosion Rate (mmpy)	Surface Coverage (θ)	Inhibition Efficiency (IE %)	Corrosion Rate (mmpy)	Surface Coverage (θ)	Inhibition Efficiency (IE %)
303K	Blank	19.838	-	-	261.685	-	-
	0.1%	11.590	0.4157	41.57	35.998	0.86247	86.24
	0.2%	9.807	0.5056	50.56	21.064	0.9195	91.95
	0.3%	6.129	0.6910	69.10	8.804	0.9663	96.63
	0.4%	4.569	0.7696	76.96	6.575	0.9748	97.48
	0.5%	4.123	0.7921	79.21	4.123	0.9842	98.42
353K	Blank	780.04	-	-	1739.851	-	-
	0.1%	474.660	0.3914	39.14	965.606	0.4450	44.50
	0.2%	418.940	0.4624	46.24	625.236	0.6406	64. 06
	0.3%	367.340	0.5290	52.90	535.296	0.6923	69.23
	0.4%	269.598	0.6543	65.43	258.676	0.8513	85.13
	0.5%	249.871	0.6793	67.93	65.309	0.9624	96.24
403K	Blank	958.807	-	-	2050.575	-	-
	0.1%	624.679	0.3484	34.84	1559.000	0.2396	23.96
	0.2%	557.586	0.4184	41.84	1430.800	0.3022	30.22
	0.3%	513.006	0.4649	46.49	909.435	0.5564	55.64
	0.4%	410.471	0.5718	57.18	819.940	0.6001	60.01
	0.5%	377.036	0.6067	60.67	428.638	0.7909	79.09

Table II

Calculated values of activation energy Ea (KJ/mole), Free energy of adsorption ΔG_{ads} (KJ / mole), enthalpy of adsorption ΔH (KJ /mole) and entropy of adsorption ΔS (KJ /mole^{-1} K^{-1}) for mild steel corrosion in 1.11N HCl and 1.12 N H_2SO_4 with S-AITO.

Inhibitor + Acid	Concentration of Inhibitor (%)	Ea (KJ /mole)	$-\Delta G_{ads}$ (KJ / mole)			ΔH (KJ/ mole)	ΔS (KJ/mole^{-1}K^{-1})
			At 303K	At 353K	At 403K		
S –AITO + 1.11N HCl	Blank	39.37	-	-	-	36.44	-
	0.1%	40.48	15.06	17.24	19.07	37.54	0.1532
	0.2%	41.02	14.28	16.06	17.74	38.09	0.1548
	0.3%	44.95	15.17	15.66	17.01	42.01	0.1641
	0.4%	45.67	15.46	16.34	17.49	42.73	0.1673
	0.5%	45.85	15.23	16.02	17.23	42.91	0.1675
S-AITO + 1.12 N H_2SO_4	Blank	20.90	-	-	-	17.96	-
	0.1%	38.26	20.54	17.89	17.30	35.32	0.1526
	0.2%	42.83	20.30	18.20	16.04	39.89	0.1643
	0.3%	47.09	21.60	17.70	18.25	44.15	0.1794
	0.4%	47.15	21.63	19.59	17.88	44.22	0.1862
	0.5%	49.00	22.27	23.33	20.23	46.06	0.1874

Table III

Potentiostatic polarization parameters for S-AITO in 1.11N HCl and 1.12N H2SO4

Conc. of inhibitor (%)	E_{corr} Vs SCE (mV)	I_{corr} $\mu A/cm^2$	Tafel slopes (mV / decade)		IE%	LPR Rp (ohm cm^2)
			ba	- bc		
302 K						
1.11N HCl						
Blank	-510	700	50	150	-	23.26
0.1 S-AITO	-549	384	49	147	45.14	41.56
0.2 S-AITO	-540	292	49	143	58.28	54.27
0.3 S-AITO	-538	205	47	145	70.71	75.18
0.4 S-AITO	-534	139	44	140	80.14	105.73
0.5 S-AITO	-530	86	43	139	87.71	165.81
1.12N H$_2$SO$_4$						
Blank	-510	700	50	150	-	23.26
0.1 S-AITO	-588	273	47	145	61.00	56.46
0.2 S-AITO	-570	228	44	143	67.43	64.08
0.3 S-AITO	-563	160	40	137	77.14	84.02
0.4 S-AITO	-552	88	37	135	87.43	143.29
0.5 S-AITO	-540	60	35	130	91.43	199.56
353 K						
1.11N HCl						
Blank	-505	1000	30	175	-	11.20
0.1 S-AITO	-530	568	28	170	43.20	18.38
0.2 S-AITO	-527	441	28	165	55.90	23.57
0.3 S-AITO	-520	380	27	160	62.00	26.40
0.4 S-AITO	-518	239	26	157	76.10	40.53
0.5 S-AITO	-510	150	23	155	85.00	57.98
1.12N H$_2$SO$_4$						
Blank	-505	1000	30	175	-	11.20
0.1 S-AITO	-572	435	30	166	56.50	25.36
0.2 S-AITO	-560	359	28	163	64.10	28.90
0.3 S-AITO	-553	279	27	160	72.10	35.95

0.4 S-AITO	-549	144	25	158	85.56	65.09
0.5 S-AITO	-540	125	24	155	87.50	72.19
333 K						
1.11N HCl						
Blank	-483	3000	75	190	-	7.78
0.1 S-AITO	-520	1823	75	185	39.23	12.71
0.2 S-AITO	-512	1403	72	182	53.23	16.63
0.3 S-AITO	-507	1214	72	180	59.53	18.39
0.4 S-AITO	-502	806	70	174	73.13	26.89
0.5 S-AITO	-496	493	68	170	83.57	42.98
1.12N H_2SO_4						
Blank	-483	3000	75	190	-	7.78
0.1 S-AITO	-540	1365	70	186	54.50	16.18
0.2 S-AITO	-543	1149	70	183	61.70	19.13
0.3 S-AITO	-530	1029	68	180	65.70	20.83
0.4 S-AITO	-523	579	66	174	80.70	35.88
0.5 S-AITO	-510	460	65	170	84.67	44.88

Table IV

A.C. impedance parameters for S-AITO in 1.11N HCl and 1.12N H2SO$_4$

Conc. of inhibitor (%)	R_{ct} (ohm cm^2)	C_{dl} (μF/cm^2)	Inhibition efficiency (IE%)	Surface coverage (θ)
1.11N HCl				
Blank	100.12	13.20	-	-
0.1 S-AITO	188.97	10.47	47.02	0.4702
0.2 S-AITO	250.62	8.45	60.05	0.6005
0.3 S-AITO	358.87	6.24	72.10	0.7210
0.4 S-AITO	558.57	4.16	82.21	0.8221
0.5 S-AITO	920.42	3.91	89.12	0.8912
1.12N H$_2$SO$_4$				
Blank	100.12	13.20	-	-
0.1 S-AITO	270.92	8.00	63.04	0.6304
0.2 S-AITO	325.62	6.99	69.25	0.6925
0.3 S-AITO	480.12	5.11	79.15	0.7915
0.4 S-AITO	922.14	2.73	89.14	0.8914
0.5 S-AITO	1672.57	1.56	94.01	0.9401
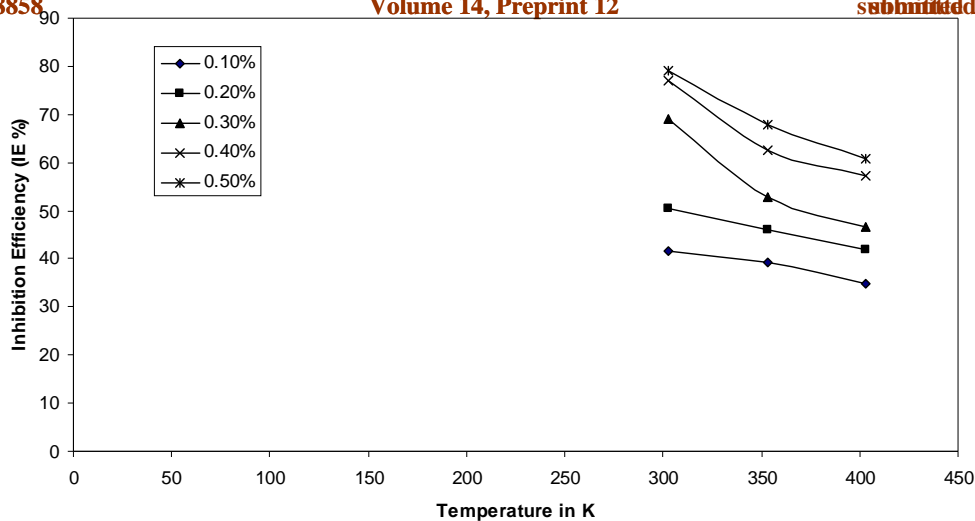
Fig.1 Relation between the inhibition efficiency and temperature for mild steel corrosion in
1.11N HCl with S-AITO
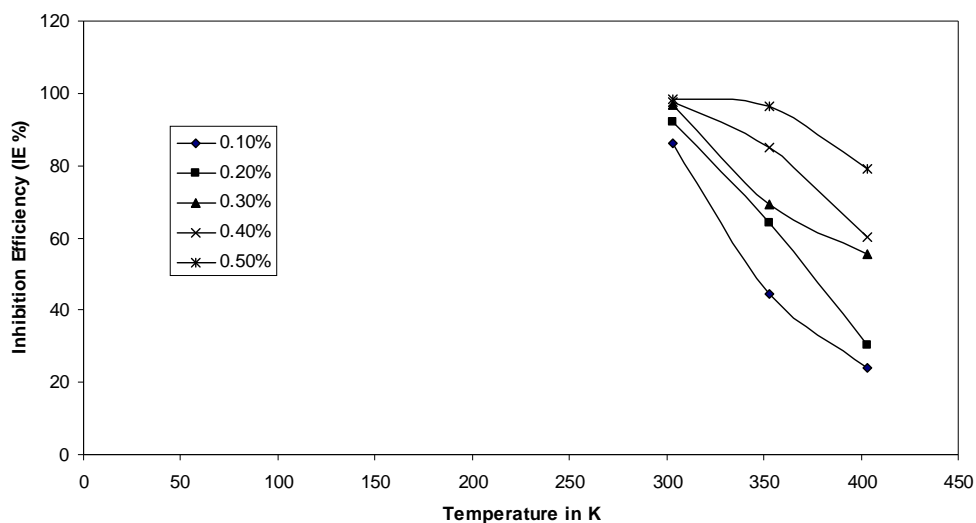
Fig. 2 Relation between the inhibition efficiency and temperature for mild steel corrosion in
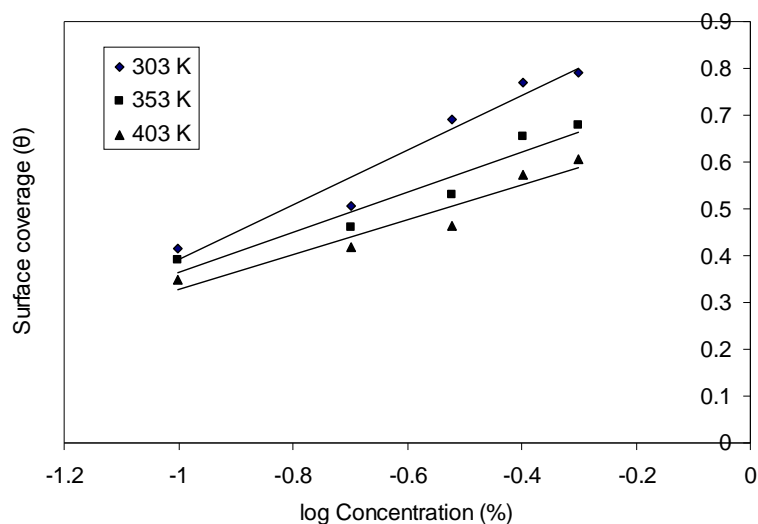1.12N H_2SO_4 with S-AITO.

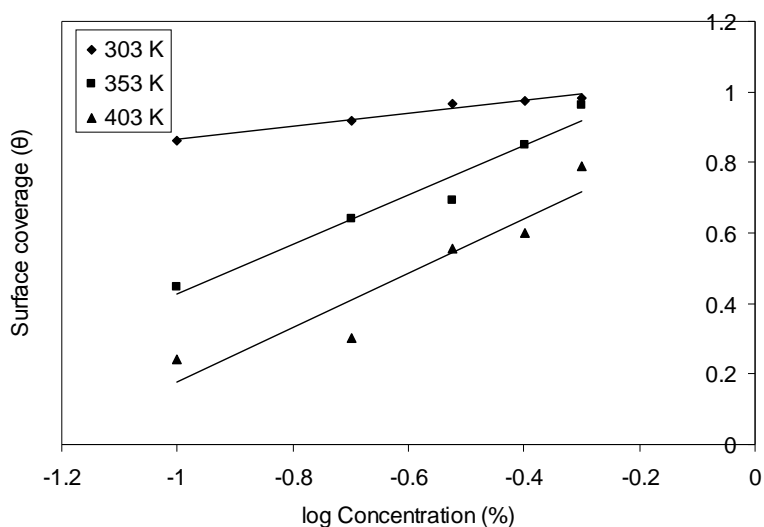Fig. 3 Temkin's adsorption isotherm plot for 1.11N HCl with S-AITO
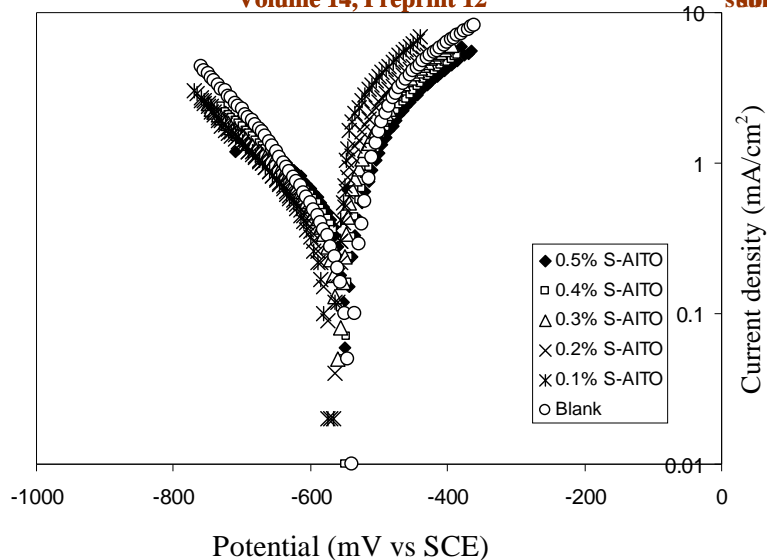

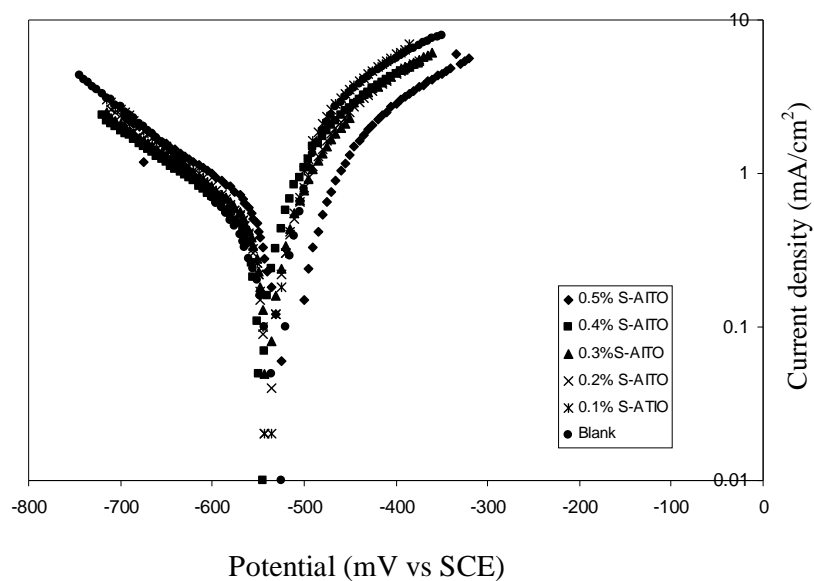Fig. 4 Temkin's adsorption isotherm plot for 1.12N H_2SO_4

Fig.5 for 1.11N HCl

Fig. 5 for 1.12N H$_2$SO$_4$

Figures 5 and 6 Typical potentiostatic curves for mild steel in 1.11N HCl and 1.12N H$_2$SO$_4$ with S-AITO at 303K
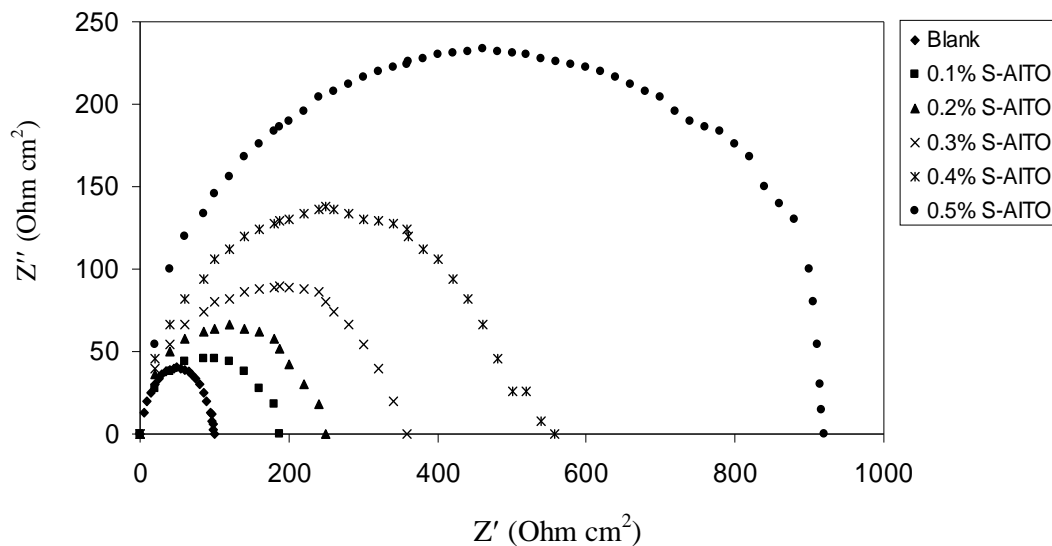
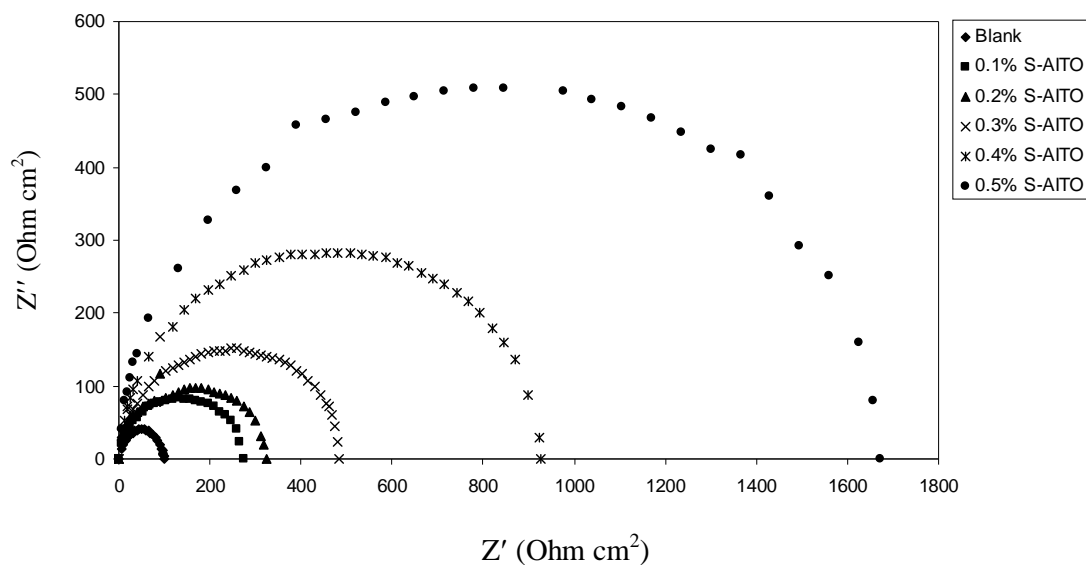Figure 7 Nyquist plots for mild steel in 1.11N HCl with S-AITO

Figure 8 Nyquist plots for mild steel in 1.12N H2SO4 with S-AITO