Effect of S-*AITA* on Mild Steel Corrosion in Acidic Medium

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S-Acetyl Isothiourea Acetate (S-AITA) 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-AITA was found to be 99.95% (0.5% of S-AITA) 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-AITA 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.*

1. 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 1.11 N HCl and 1.12N H₂SO₄ solutions in the absence and presence of S-AITA 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-AITA. The adsorption characteristic of S-AITA was studied in order to across the adsorption isotherm that the experiment data can fit and some thermodynamic parameters of adsorption calculated.

2. Experimental

2.1 Weight loss measurement

Mild steel specimens were cut to the size of 5 cm x 1 cm 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 1.11 N HCl and 1.12 N H₂SO₄ acids with inhibitor S-Acetyl Isothiourea Acetate (S-AITA) 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 a compound known as S-acetylthiuronium chloride or S-acetyl isothiourea hydrochloride. On mixing a solution of the alkali salt of carboxylic acid (sodium acetate), the S-Acetyl Isothiourea Acetate is formed rapidly. Synthesized com-

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pound was characterized through the spectral data and the purity was confirmed by thin layer chromatography (TLC). The structure of S-AITA is

$$\left(\begin{array}{ccccccc} & O & & NH_2 & \\ & \| & & \| & \\ CH3 & - & C & - & S & - & C & - & NH_2 \end{array} \right)^+ CH_3COO^-$$
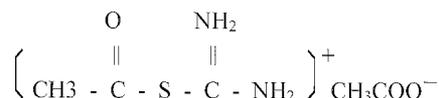
2.2 Potentiostatic polarization /A.C. impedance measurement

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. 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.

3. Results and discussion

3.1 Weight loss measurements

Table 1 shows the values of inhibition efficiency (IE %), surface coverage (θ) and corrosion rate obtained for different concentrations of inhibitor (S-AITA) in 1.11 N HCl and 1.12 N 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)]

$$\text{Inhibition efficiency (IE \%)} = \frac{Wu - Wi}{Wu} \times 100 \quad (1)$$

$$\text{Surface Coverage } (\theta) = \frac{Wu - Wi}{Wu} \quad (2)$$

Where Wu and Wi are the corrosion rates for mild steel in the absence and presence of inhibitor (S-AITA) res

Table 1. Calculated values of corrosion rate, inhibition efficiency and surface coverage for S AITA in 1.11 N HCl and1.12 N H$_2$SO$_4$ from weight loss method

| Temp.	Conc.of S-AITA (%)	1.11 N HCl			1.12 N H$_2$SO$_4$		
		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%	7.133	0.6406	64.06	7.578	0.9710	97.10
	0.2%	6.464	0.6741	67.41	6.018	0.9770	97.70
	0.3%	5.461	0.7247	72.47	5.238	0.9799	97.99
	0.4%	4.123	0.7921	79.21	4.792	0.9816	98.16
	0.5%	2.897	0.8539	85.39	0.111	0.9995	99.95
353K	Blank	780.040	-	-	1739.85	-	-
	0.1%	307.825	0.6053	60.53	115.574	0.9259	92.59
	0.2%	289.770	0.6285	62.85	108.218	0.9378	93.78
	0.3%	242.181	0.6895	68.95	91.500	0.9474	94.74
	0.4%	193.477	0.7519	75.19	56.170	0.9677	96.77
	0.5%	160.377	0.7943	79.43	55.52	0.9680	96.80
403K	Blank	958.807	-	-	2050.575	-	-
	0.1%	400.441	0.5823	58.23	229.476	0.8880	88.80
	0.2%	374.473	0.5647	56.47	157.590	0.9231	92.31
	0.3%	308.048	0.6787	67.87	143.436	0.9300	93.00
	0.4%	260.459	0.7283	72.83	138.755	0.9323	93.23
	0.5%	248.534	0.7407	74.07	95.067	0.9536	95.36
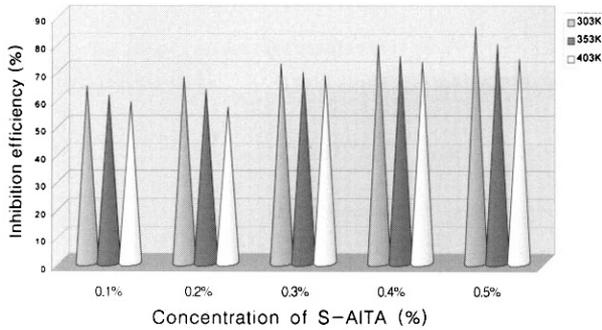
Fig. 1
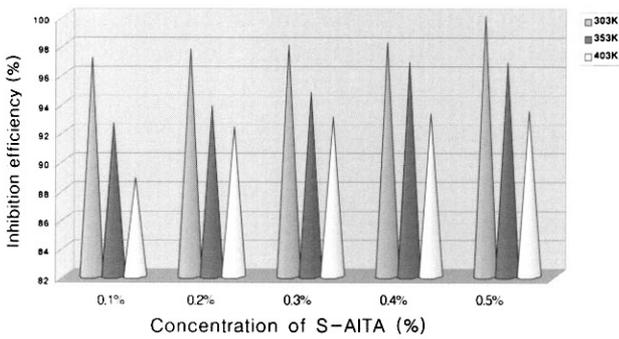
Fig. 2

Fig. 1 and 2. Relation between the inhibition efficiency and temperature for mild steel corrosion in 1.11 N HCl and 1.12 N H_2SO_4 with S-AITA.

pectively at the same temperature.

It clearly indicates that the addition of inhibitor to the

acids (1.11N HCl and 1.12N H_2SO_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 1.11 N HCl and 1.12 N H_2SO_4. 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 inhibitor efficiency of S-AITA was 85.39% in 1.11 N hydrochloric acid and 99.95% in 1.12 N sulphuric acid at 0.5% (S-AITA). 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.

3.2 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 (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_2SO_4 with and without inhibitor. Energy of activation (Ea) has been calculated from Arrhenius equation.[20)-22)]

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

Table 2. 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_2SO_4 with S-AITA.

Inhibitor + Acid	Concentration of Inhibitor (%)	Ea (KJ/mole)	$_{ads}$ (KJ / mole)			ΔH (KJ / mole)	ΔS (KJ/mole^{-1} K^{-1})
			At 303K	At 353K	At 403K		
S-AITA + 1.11 N HCl	Blank	39.37	-	-	-	36.44	-
	0.1%	40.89	17.37	19.80	22.28	37.96	0.0514
	0.2%	40.94	16.00	18.05	19.72	38.01	0.0569
	0.3%	41.21	15.58	17.66	19.99	38.28	0.5817
	0.4%	42.09	15.79	17.73	19.83	39.16	0.0606
	0.5%	45.20	16.31	17.78	19.29	42.26	0.0693
S-AITA + 1.12 N H_2SO_4	Blank	20.90	-	-	-	17.96	-
	0.1%	33.15	24.76	25.95	28.11	30.22	0.0112
	0.2%	33.60	23.62	24.47	27.17	30.67	0.0158
	0.3%	34.17	22.94	23.80	26.16	31.24	0.0196
	0.4%	34.62	22.44	24.45	25.31	31.69	0.0216
	0.5%	68.53	31.01	23.82	25.90	65.598	0.1096

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 1.11 N HCl and 1.12 N H_2SO_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 inhibitors 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]

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

and K is given by

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

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

$$\Delta H = Ea - RT \tag{6}$$

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

ΔS can be easily calculated at 303K to 403K for the different concentration of S-AITA. 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.

3.3 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, $H_2O_{(S)}$

$$Org_{(aq)} + x\ H_2O_{(s)} = Org_{(s)} + x\ (H_2O)_{(aq)} \tag{8}$$
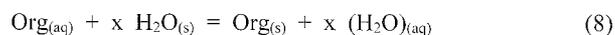
Where x is the size ratio, is the number of water molecules displaced by one molecule of organic inhibitors.

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.

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

Where $f\ (\theta, x)$ 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-AITA.

3.4 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 1.11 N HCl and 1.12 N H_2SO_4 at 303 K for S-AITA. Similar curves were also obtained for 1.11 N HCl and 1.12 N 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-AITA brings 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

Table 3. Potentiostatic polarization parameters for S-AITA in 1.11 N HCl and 1.12 N H₂SO₄

Conc. of inhibitor (%)	E_{corr} Vs SCE (mV)	I_{corr} $\mu A/cm^2$	Tafel slopes (mV / decade)		IE%	LPR Rp (ohm cm²)
			ba	-bc		
303 K						
1.11N HCl						
Blank		210	50	130	-	74.67
0.1 S-AITA		80	50	127	61.90	194.72
0.2 S-AITA		64	47	120	69.52	216.18
0.3 S-AITA		53	46	122	74.76	273.68
0.4 S-AITA		42	50	116	80.00	361.22
0.5 S-AITA		14	44	110	93.33	974.77
1.12N H₂SO₄						
Blank		210	50	130	-	74.67
0.1 S-AITA		64	40	124	69.52	205.19
0.2 S-AITA		47	40	120	77.62	277.16
0.3 S-AITA		28	38	116	86.67	443.88
0.4 S-AITA		21	37	114	90.00	577.58
0.5 S-AITA		6	34	110	97.14	1879.59
353 K						
1.11N HCl						
Blank		680	100	150	-	38.31
0.1 S-AITA		280	95	145	58.82	89.01
0.2 S-AITA		231	93	140	66.03	105.04
0.3 S-AITA		184	90	136	72.94	127.81
0.4 S-AITA		128	85	130	81.18	174.35
0.5 S-AITA		64	80	125	90.59	330.96
1.12N H₂SO₄						
Blank		680	100	150	-	38.31
0.1 S-AITA		221	92	144	67.43	110.29
0.2 S-AITA		175	90	140	74.21	135.93
0.3 S-AITA		112	86	138	83.50	205.41
0.4 S-AITA		85	83	136	87.21	263.31
0.5 S-AITA		42	80	133	93.82	516.44
403 K						
1.11N HCl						
Blank		1400	150	150	-	23.26
0.1 S-AITA		650	140	145	53.57	47.58
0.2 S-AITA		509	135	140	63.64	58.63
0.3 S-AITA		412	130	137	70.57	70.30
0.4 S-AITA		333	125	132	76.21	83.72
0.5 S-AITA		203	120	130	85.50	133.47
1.12N H₂SO₄						
Blank		1400	150	150	-	23.26
0.1 S-AITA		519	150	142	62.94	61.03
0.2 S-AITA		455	147	138	67.50	67.93
0.3 S-AITA		294	147	135	78.97	103.93
0.4 S-AITA		198	145	130	85.88	150.32
0.5 S-AITA		169	140	132	87.92	174.56

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 polari-zation 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 inhi-
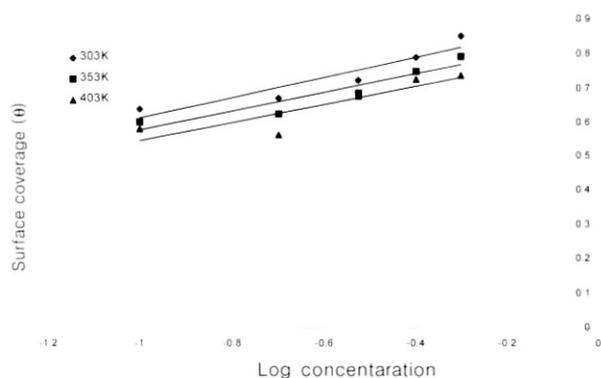
Fig. 3. Temkin's adsorption isotherm plot for 1.11 N HCl with S-AITA
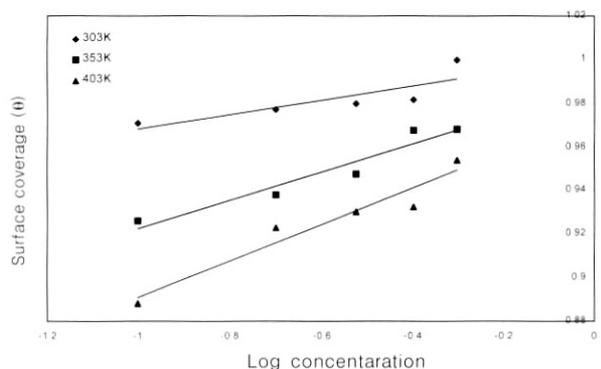

Fig. 4. Temkin's adsorption isotherm plot for 1.12 N H$_2$SO$_4$ with S-AITA

Fig. 5. for 1.11 N HCl

Fig. 6. for 1.12 N H$_2$SO$_4$

Figs. 5. and 6. Typical potentiostatic curves for mild steel in 1.11 N HCl and 1.12 N H$_2$SO$_4$ with S-AITA at 303K

bition 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.

3.5 Impedance measurements

A.C. impedance measurements were carried out at room temperature for corrosion of mild steel in 1.11 N HCl and 1.12 N H$_2$SO$_4$ 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$_2$SO$_4$ 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.
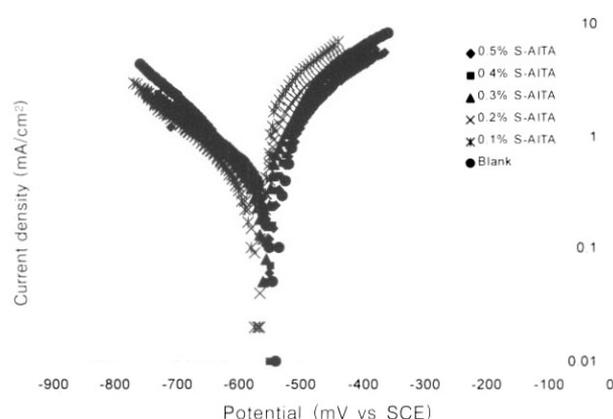
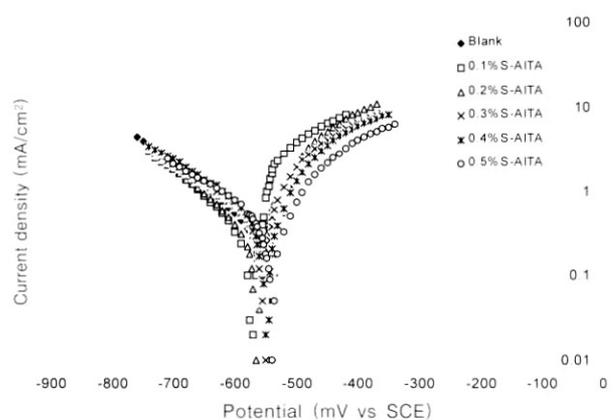**Table 4.** A.C. impedance parameters for S-AITA in 1.11 N HCl and 1.12 N H$_2$SO$_4$

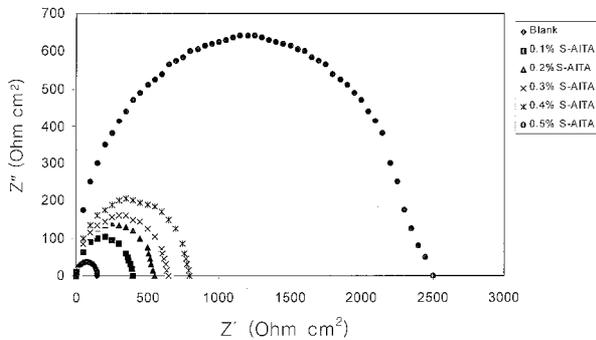| Conc. of inhibitor (%) | 1 N H$_3$PO$_4$ | | |
	R$_{ct}$ (ohm cm^2)	C$_{dl}$ (μF/cm^2)	Inhibition efficiency (IE%)
1.11N HCl			
Blank	150.60	5.74	-
0.1 S-AITA	407.10	2.71	63.00
0.2 S-AITA	538.15	2.19	72.01
0.3 S-AITA	630.42	1.98	76.11
0.4 S-AITA	793.15	1.67	81.01
0.5 S-AITA	2550.75	0.54	94.10
1.12N H$_2$SO$_4$			
Blank	150.60	5.74	-
0.1 S-AITA	520.18	2.21	71.05
0.2 S-AITA	685.18	1.80	78.02
0.3 S-AITA	1300.17	1.01	88.42
0.4 S-AITA	1910.68	0.73	92.12
0.5 S-AITA	7370.33	0.19	97.96

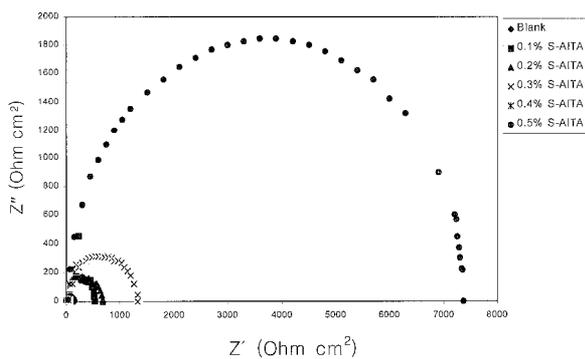**Fig. 7.** Nyquist plots for mild steel in 1.11 N HCl with S-AITA

Fig. 8. Nyquist plots for mild steel in 1.12 N H₂SO₄ with S-AITA

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]

4. Conclusions

The conclusions reported in this paper are the following

1. S-AITA acted as efficient corrosion inhibitor in 1.11 N HCl and 1.12 N H₂SO₄.

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

3. The maximum inhibition efficiency of S-AITA was 85.39% in 1.11 N HCl and 99.95% in 1.12 N H₂SO₄ (0.5% of S-AITA).

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-AITA 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-AITA acted as mixed type inhibitor.

Acknowledgments

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