

Corrosion Inhibitory Effects of Some Substituted Thiourea on Mild Steel in Acid Media

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Abstract

Mass loss and thermometric methods have been used to study the inhibition of mild steel corrosion in HCl and H₂SO₄ solution by the pyridyl substituted thiourea compound 1-(2,6-diazene)-3-benzyl thiourea (ST₁), 1-(3'-pyridyl) – 3 – benzyl thiourea (ST₂), 1 – (3' - pyridyl) – 1 –phenyl thiourea (ST₃), 1-(2' - pyridyl)-3-phenyl thiourea (ST₄). Values of inhibition efficiency obtained from the two methods are in good agreement with each other and are dependent upon the concentration of inhibitor and acid. The difference in the inhibition behaviour of the compounds have been explained in terms of the solubility of the substituted thiourea compounds and strength of the inhibitor-metal bond. Inhibition efficiency of all inhibitors increases with increasing concentration of inhibitor. Inhibition efficiency is more in case of H₂SO₄ rather than in HCl. Inhibition efficiency was found maximum upto 99.26% for mild steel in H₂SO₄ solution. Inhibition efficiencies of synthesised substituted thiourea have been found much more than their parent thiourea.

Keywords: Corrosion inhibition, weight loss method, thermometric method, surface coverage, corrosion rate etc.

Introduction

Mild steel finds a variety of applications industrially, for mechanical and structural purposes, like bridge work, building, boiler plates, steam engine parts and automobiles. It finds various uses in most of the chemical industries due to its low cost and easy availability for fabrication of various reaction vessels, tanks, pipes etc. Since it suffers from severe corrosion in aggressive environment, it has to be protected acids like HCl and H₂SO₄ have been used for drilling operations, pickling baths and in decaling processes.

Corrosion of mild steel and its alloys in different acid media have been extensively studied¹⁻⁴. It has been reported⁵⁻⁶ that addition of certain organic compounds bearing hetero atoms, retards the corrosion of mild steel in acidic environments. Recently considerable interest has been generated in the use of nitrogen, oxygen and sulphur containing organic compounds as corrosion inhibitor for mild steel in different acids⁷⁻⁸.

Organic compounds having hetero atoms like O, N, S and in some cases Se, are found to have higher basicity and electron density. Thus to help corrosion inhibition⁹, O, N and S are the active centres for the process of adsorption on the metal surface¹⁰⁻¹⁵. The electric charge, orientation, shape and size of the molecule play an important role on the effectiveness of inhibition. Efficiency of their inhibitory effect depends upon the basicity of these atoms¹⁶⁻¹⁸.

Among these, thiourea and its derivatives have been investigated extensively. These are polar molecules in which

S atom having permanent –ve charge and N atom have +ve charge. As the molecule approaches the electrode surface the electric field of double layer increases the polarization of molecule and induces additional charges on S and N atoms, a condition that enhance the adsorption of molecule.

In the present study an attempt has been made to study the influence of varying concentration of substituted thiourea viz. (ST₁, ST₂, ST₃, ST₄) on corrosion of mild steel in different concentration of hydrochloric acid, sulphuric acid employing mass loss and thermometric method.

Material and Methods

Mild steel specimens of composition 99.3% Fe, 0.2% C, 0.3% Mg, 0.14% Si and 0.04 % S of size 2.00 cm × 2.00 cm × 0.03 cm were used for the complete immersion test. All the specimens were polished by buffing and rubbing with emery paper to obtain mirror like finishing. The solution of HCl were prepared by using double distilled water. All chemicals used were of AR grade.

Each specimen was suspended by a V-shaped glass hook made by capillary tubes and immersed in a glass beaker containing 50 mL of the test solution at room temperature, after the test specimen was cleaned with benzene and then dried with hot air dryer. The percentage efficiency was calculated as¹⁹

$$\eta \% = 100 \frac{(\Delta W_u - \Delta W_i)}{\Delta W_u}$$

where ΔW_u and ΔW_i are the weight loss of the metal in uninhibited and inhibited solution, respectively. Inhibition efficiency were also calculated using a thermometric method. This involve the immersion of single specimen of measurement $2.00 \times 2.00 \times 0.03$ cm in a reaction insulating chamber having 50 mL of solution at an initial room temperature. Temperature changes were measured at regular intervals using a thermometer with a precision of 0.1°C . The increase in temperature was initially slow then rapid and attained a maximum value and then decreased. The maximum temperature was noted. Percentage inhibition efficiency was calculated as²⁰

$$\eta \% = \frac{100 (RN_f - RN_i)}{RN_f}$$

where RN_f and RN_i are the reaction number in the free solution and in presence of inhibitor. RN is defined as

$$RN = \frac{(T_m - T_i)}{t}$$

where T_m and T_i are maximum and initial temperature, respectively and t is the time in minutes required to attain maximum temperature. The corrosion rate (CR) in mm/yr can be obtained by the following equation²¹

$$\text{Corrosion rate (mm / yr)} = \frac{\Delta W \times 87.6}{A \times T \times d}$$

where, ΔW is weight loss in mg, A is area of specimen in cm^2 , T is time of exposure in hours, d is density of metal in gm/cm^3 (θ) can be calculated as²²

$$\theta = \frac{(\Delta W_u - \Delta W_i)}{\Delta W_u}$$

where ΔW_u and ΔW_i are the weight loss of the metal in uninhibited and in inhibited solution, respectively.

Results and Discussion

Weight Loss Method: Weight loss, percentage inhibition efficiencies, corrosion rate and surface coverage for different concentration of HCl and inhibitor are given in table-1 and for different concentration of H_2SO_4 and inhibitors are given in table-2. It can be seen from the both the tables that inhibition efficiency of inhibitor increases with increasing concentration of inhibitor. Inhibition efficiency also increases with increasing concentration of acid and all the inhibitors show maximum inhibition efficiency at the highest concentration of acids used i.e. 2.5 N HCl and 2.5 NH_2SO_4 . The maximum inhibition efficiency was obtained for (ST₁) at an inhibitor concentration of 0.8% in 2.5 N HCl and 2.5 N H_2SO_4 i.e. 98.48% and 99.26%, respectively. These results show that substituted thiourea show more inhibition efficiency in H_2SO_4 than in HCl. The variation of percentage inhibition efficiency with inhibitor concentration are depicted graphically in figure 1 for HCl and in figure 2 for H_2SO_4 . Figures show a linear curve of percentage inhibition efficiency with the concentration of inhibitor, indicating that the inhibition efficiency increases with increasing inhibitor concentration.

Thermometric Method: Inhibition efficiency values were also determined by the thermometric method. Temperature changes for mild steel in 2N, 3N and 4N sulphuric acid and hydrochloric acid solution were recorded with various inhibitor concentration. However no significant temperature changes were measured in the acid concentration of 1N HCl and H_2SO_4 solution. Results obtained are depicted in table 3 for HCl and table - 4 for H_2SO_4 . The results obtained are in good agreement with those from weight loss experiments. Inhibition efficiency increases with increasing acid concentration. The variation of reaction number with inhibitor concentration, presented graphically in figure 3 for HCl and in figure 4 for H_2SO_4 shows linear behaviour with positive slope, indicating that the reaction number increase with increasing acid concentration it decreases with increasing inhibitor concentration. Generally, the Inhibitor having oxygen, nitrogen and sulphur atoms responsible for the adsorption on metallic surface. This process may block active sides, hence may decrease the corrosion rate. Adsorption plays an important role in the inhibition of metallic corrosion by organic inhibitors. Many investigators have used the Langmuir adsorption isotherm to study inhibitor characteristics. Assuming that the inhibitors adsorbed on the metal surface decrease the surface area available for cathodic and anodic reactions to take place, Hoar and Holliday have shown that the Langmuir isotherm

$$\log [\theta / (1 - \theta)] = \log A + \log C - (\theta / 2.3 RT)]$$

should give a straight line of unit gradient for the plot of $\log[\theta / (1 - \theta)]$ versus $\log C$, where surface coverage θ is calculated as $(RN_f - RN) / RN_f$. A is a temperature independent constant, and C is the bulk concentration of the inhibitor (mol L^{-1})

The corresponding plots, shown in figure 5 and 6 are linear, but the gradients are not equal to unity as would be expected for the ideal Langmuir adsorption isotherm equation.

It has also been observed that the efficiency is higher in higher concentration of HCl and H_2SO_4 . This may be because of the fact that the inhibitor ionize more readily under more acid strength and is absorbed more easily on the surface of metal. Acids which have more dissociation constant i.e. higher values of K_a or lower values of $\text{p}K_a$ like HCl and H_2SO_4 enhance the ionization of thiourea thus causes more adsorption of substituted thiourea on metal surface. Therefore they act as better inhibitor at higher concentrations. Adsorption plays an important role in the inhibition of metallic corrosion by organic inhibitors. The efficiencies of inhibitors expressed as the relative reduction in corrosion rate can be quantitatively related to the amount of adsorbed inhibitors on the metal surface. It is assumed, that the corrosion reaction are prevented from occurring over the active sites of the metal surface covered by adsorbed inhibitors species, whereas the corrosion reaction occurs normally on the surface at inhibitors free area.

TABLE – 1
Weight loss and percentage inhibition efficiency (η %) for mild steel in HCl solution with given inhibitor additions

Inhibitor Addition	1.0 N HCl (24 hr)				1.5 N HCl (24 hr)			
	ΔM (mg)	I.E. (η %)	Corrosion rate(mmpy)	Surface Coverage (θ)	ΔM (mg)	I.E. (η %)	Corrosion rate(mmpy)	Surface Coverage (θ)
Uninhibited	–	–	–	–	–	–	–	–
ST ₁								
0.2%	68	89.95	5.39	0.8995	49	92.57	3.88	0.9257
0.4%	62	90.84	4.91	0.9084	48	92.72	3.80	0.9272
0.6%	52	92.31	4.12	0.9231	41	93.78	3.25	0.9378
0.8%	48	92.90	3.80	0.9290	35	94.69	2.77	0.9469
ST ₂								
0.2%	72	89.36	5.70	0.8936	50	92.42	3.96	0.9242
0.4%	68	89.95	5.39	0.8995	49	92.57	3.87	0.9257
0.6%	55	91.87	4.36	0.9187	45	93.18	3.56	0.9318
0.8%	52	92.31	4.12	0.9231	40	93.93	3.17	0.9393
ST ₃								
0.2%	75	88.92	5.94	0.8892	55	91.66	4.36	0.9166
0.4%	70	89.66	5.55	0.8966	50	92.42	3.96	0.9242
0.6%	60	91.13	4.75	0.9113	48	92.72	3.80	0.9272
0.8%	59	91.28	4.67	0.9128	45	93.18	3.56	0.9318
ST ₄								
0.2%	80	24.91	6.34	0.2491	55	54.66	4.36	0.5466
0.4%	78	59.96	6.18	0.5996	51	75.42	4.04	0.7542
0.6%	70	66.94	5.56	0.6694	50	85.90	3.96	0.8590
0.8%	70	76.07	5.55	0.7607	45	90.50	3.56	0.9050
Inhibitor Addition	2N HCl (3 hr.)				2.5 N HCl (3 hr.)			
	ΔM (mg)	I.E. (η %)	Corrosion rate(mmpy)	Surface Coverage (θ)	ΔM (mg)	I.E. (η %)	Corrosion rate(mmpy)	Surface Coverage (θ)
Uninhibited	–	–	–	–	–	–	–	–
ST ₁								
0.2%	40	93.30	25.36	0.9330	35	94.10	22.19	0.9410
0.4%	39	93.62	24.72	0.9362	30	94.94	19.02	0.9494
0.6%	30	95.09	19.02	0.9509	16	97.47	10.14	0.9747
0.8%	15	97.54	09.51	0.9754	9	98.48	5.706	0.9848
ST ₂								
0.2%	41	93.30	25.99	0.9330	40	93.26	25.36	0.9326
0.4%	40	93.46	25.36	0.9346	39	93.43	24.72	0.9343
0.6%	32	94.77	20.28	0.9477	25	95.79	15.85	0.9579
0.8%	19	96.89	12.04	0.9689	11	98.14	6.974	0.9814
ST ₃								
0.2%	50	91.83	31.70	0.9183	45	92.42	28.53	0.9242
0.4%	34	94.44	21.55	0.9444	44	92.59	26.13	0.9449
0.6%	28	95.42	17.75	0.9542	28	95.28	17.75	0.9528
0.8%	20	96.73	12.68	0.9673	11	98.14	6.974	0.9814
ST ₄								
0.2%	56	90.84	35.28	0.9084	52	91.24	32.96	0.9175
0.4%	30	93.62	19.02	0.9362	50	91.58	31.70	0.9461
0.6%	26	95.75	16.48	0.9575	32	94.61	19.00	0.9730
0.8%	23	96.24	14.58	0.9624	16	97.97	10.14	0.9797

TABLE – 2
Weight loss and percentage inhibition efficiency (η %) for mild steel in H_2SO_4 solution with given inhibitor additions

Inhibitor Addition	1.0 N H_2SO_4 (24 hr.)				1.5 N H_2SO_4 (24 hr.)			
	ΔM (mg)	I.E. (η %)	Corrosion rate(mmpy)	Surface Coverage (θ)	ΔM (mg)	I.E. (η %)	Corrosion rate(mmpy)	Surface Coverage (θ)
Uninhibited	–	–	–	–	–	–	–	–
ST ₁								
0.2%	43	93.59	3.40	0.9359	40	94.10	3.17	0.9410
0.4%	38	94.33	3.01	0.9433	24	96.46	1.90	0.9646
0.6%	28	95.82	2.22	0.9582	20	97.05	1.58	0.9705
0.8%	15	97.76	1.18	0.9776	9	98.67	0.71	0.9867
ST ₂								
0.2%	45	93.29	3.56	0.9329	43	93.65	3.40	0.9365
0.4%	40	94.03	3.17	0.9403	27	96.01	2.14	0.9601
0.6%	29	95.67	2.29	0.9567	22	96.75	1.74	0.9675
0.8%	19	97.16	1.50	0.9716	11	98.37	0.87	0.9837
ST ₃								
0.2%	45	93.29	3.56	0.9329	45	93.36	3.56	0.9336
0.4%	41	95.38	3.25	0.9538	30	95.57	2.37	0.9557
0.6%	30	95.52	2.37	0.9552	25	96.31	1.98	0.9631
0.8%	21	97.61	1.66	0.9761	14	97.93	1.11	0.9793
ST ₄								
0.2%	48	92.84	3.80	0.9284	47	93.06	3.72	0.9306
0.4%	35	94.78	2.77	0.9478	32	94.83	2.53	0.9483
0.6%	33	95.08	2.61	0.9508	29	95.72	2.29	0.9572
0.8%	26	96.56	2.06	0.9656	15	97.78	1.18	0.9778
Inhibitor Addition	2.0 N H_2SO_4 (3 hr.)				2.5 N H_2SO_4 (3 hr.)			
	ΔM (mg)	I.E. (η %)	Corrosion rate(mmpy)	Surface Coverage (θ)	ΔM (mg)	I.E. (η %)	Corrosion rate(mmpy)	Surface Coverage (θ)
Uninhibited	–	–	–	–	–	–	–	–
ST ₁								
0.2%	27	96.02	17.11	0.9602	25	96.32	15.85	0.9632
0.4%	22	96.75	13.94	0.9675	14	97.94	8.87	0.9794
0.6%	15	97.79	9.51	0.9779	10	98.52	6.34	0.9852
0.8%	8	98.96	5.07	0.9896	5	99.26	3.17	0.9926
ST ₂								
0.2%	29	95.72	18.38	0.9572	79	95.73	15.85	0.9573
0.4%	25	96.31	15.85	0.9631	19	97.20	12.04	0.9720
0.6%	19	97.20	12.04	0.9720	14	97.94	8.87	0.9794
0.8%	8	98.96	5.07	0.9896	8	98.82	5.07	0.9882
ST ₃								
0.2%	30	95.58	19.02	0.9558	30	95.58	19.02	0.9558
0.4%	30	95.58	19.02	0.9558	19	97.20	12.04	0.9720
0.6%	20	97.05	12.68	0.9705	15	97.79	9.51	0.9779
0.8%	9	98.67	5.70	0.9867	8	98.82	5.07	0.9882
ST ₄								
0.2%	35	94.84	22.19	0.9484	33	95.14	20.92	0.9514
0.4%	32	95.28	20.28	0.9528	20	97.05	12.68	0.9705
0.6%	22	96.17	13.94	0.9617	20	97.05	12.68	0.9705
0.8%	10	98.52	6.34	0.9852	9	98.67	5.70	0.9867

TABLE – 3
Reaction Number (RN) and percentage inhibition (η %) for Mild Steel in HCl solution with given inhibitor additions

Inhibitor Addition	2N HCl		3N HCl		4N HCl	
	RN (kmin^{-1})	I.E. (η %)	RN (kmin^{-1})	I.E. (η %)	RN (kmin^{-1})	I.E. (η %)
Uninhibited	0.0277	–	0.0666	–	0.1500	–
ST ₁						
0.2%	0.0077	72.20	0.0170	74.47	0.0383	74.46
0.4%	0.0072	74.00	0.0160	75.97	0.0366	75.60
0.6%	0.0061	77.97	0.0138	79.27	0.0300	80.00
0.8%	0.0050	81.94	0.0110	83.48	0.0250	83.33
ST ₂						
0.2%	0.0100	63.89	0.0238	64.26	0.0508	66.13
0.4%	0.0088	68.23	0.0216	67.56	0.0475	68.33
0.6%	0.0083	70.03	0.0194	70.87	0.0441	70.60
0.8%	0.0061	77.97	0.0150	77.47	0.0400	73.33
ST ₃						
0.2%	0.0111	59.92	0.0250	63.03	0.0541	63.93
0.4%	0.0105	62.09	0.0244	63.36	0.0491	67.26
0.6%	0.0090	67.50	0.0211	68.31	0.0466	68.93
0.8%	0.0080	71.11	0.0200	69.96	0.0450	70.00
ST ₄						
0.2%	0.0122	55.95	0.0277	58.40	0.0566	62.26
0.4%	0.0116	58.12	0.0266	60.06	0.0525	65.00
0.6%	0.0100	63.89	0.0222	66.66	0.0500	66.66
0.8%	0.0070	74.72	0.0216	67.56	0.0483	67.80

TABLE – 4
Reaction Number (RN) and percentage inhibition (η %) for Mild Steel in H₂SO₄ solution with given inhibitor additions

Inhibitor Addition	2N H ₂ SO ₄		3N H ₂ SO ₄		4N H ₂ SO ₄	
	RN (kmin^{-1})	I.E. (η %)	RN (kmin^{-1})	I.E. (η %)	RN (kmin^{-1})	I.E. (η %)
Uninhibited	0.1388	–	0.1666	–	0.2833	–
ST ₁						
0.2%	0.0388	72.04	0.0455	72.68	0.0725	74.40
0.4%	0.0377	72.83	0.0444	73.34	0.0708	75.00
0.6%	0.0361	73.99	0.0416	75.03	0.0691	75.60
0.8%	0.0350	74.78	0.0405	75.69	0.0666	76.49
ST ₂						
0.2%	0.0416	70.02	0.0477	71.36	0.0741	73.84
0.4%	0.0405	70.82	0.0455	72.68	0.0716	74.72
0.6%	0.0380	72.62	0.0438	73.70	0.0700	75.29
0.8%	0.0372	73.19	0.0422	74.66	0.0675	76.17
ST ₃						
0.2%	0.0438	68.44	0.0483	71.00	0.0766	72.96
0.4%	0.0422	69.59	0.0461	72.32	0.0750	73.52
0.6%	0.0388	72.04	0.0444	73.34	0.0733	74.12
0.8%	0.0377	72.83	0.0427	74.36	0.0725	74.40
ST ₄						
0.2%	0.0444	68.01	0.0500	69.98	0.0858	69.71
0.4%	0.0433	68.80	0.0488	70.70	0.0833	70.59
0.6%	0.0400	71.18	0.0466	72.02	0.0791	72.07
0.8%	0.0388	72.04	0.0450	72.98	0.0758	73.24

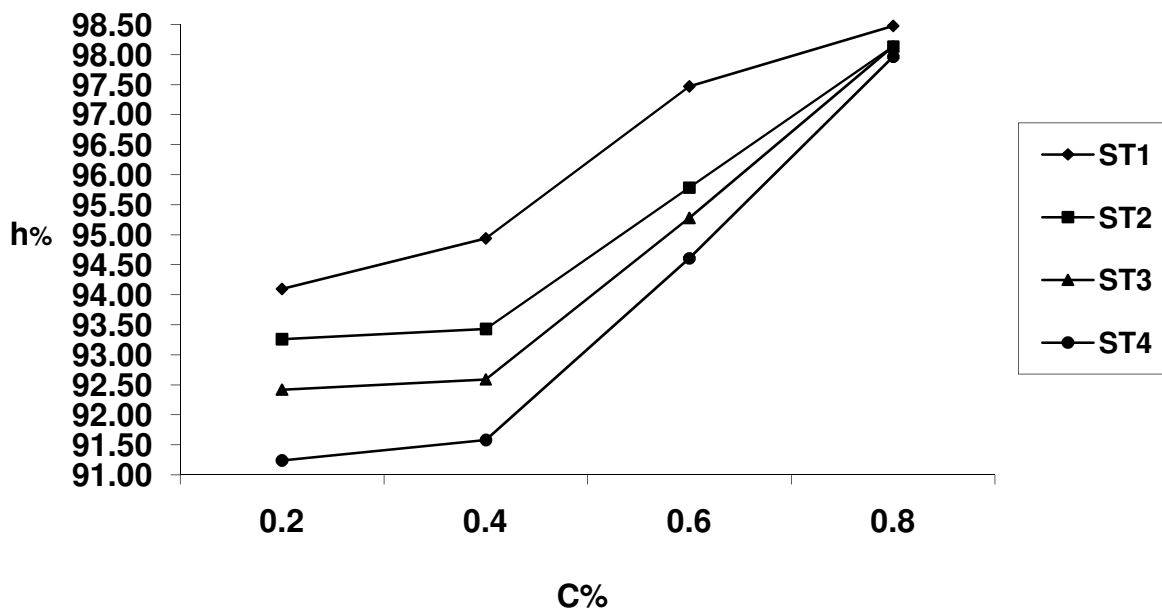


Figure-1
Variation of inhibition efficiency ($\eta\%$) with inhibitor concentration (C) for aluminium in 2.5 N HCl

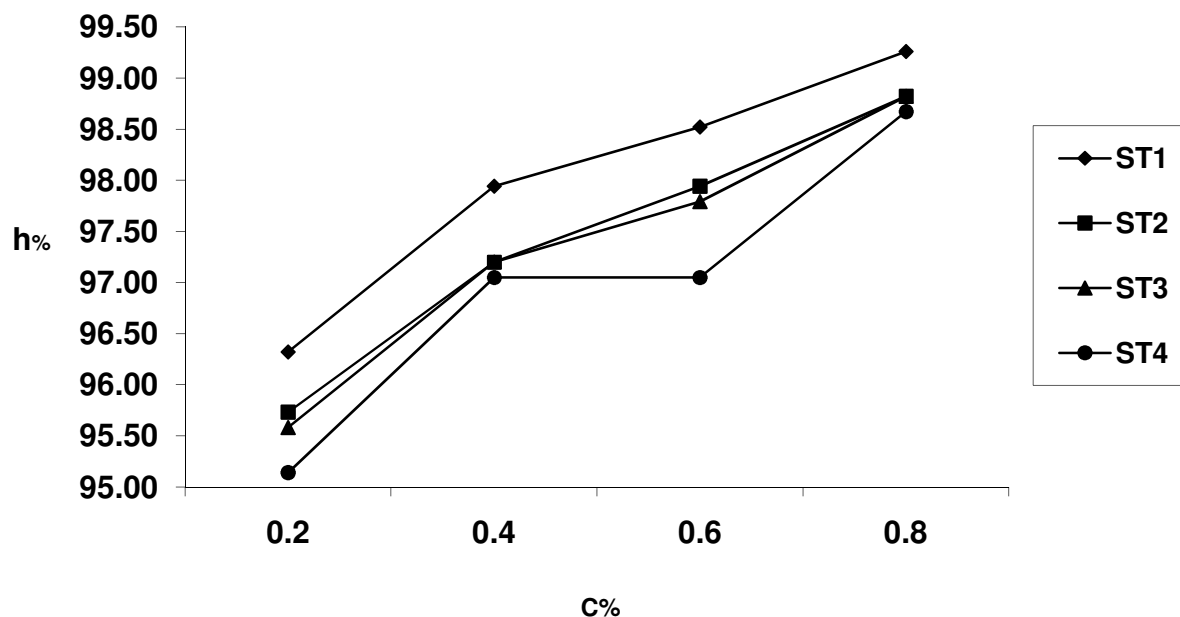


Figure-2
Variation of inhibition efficiency ($\eta\%$) with inhibitor concentration (C) for aluminium in 2.5 N H₂SO₄
ST₁: 1 - (2, 6-diazene) - 3 -benzyl thiourea, ST₂ : 1 - (3'-pyridyl) - 3 - benzyl thiourea, ST₃ : 1 - (3'- pyridyl) - 1 -phenyl thiourea, ST₄ : 1 - (2'- pyridyl) - 3 -phenyl thiourea

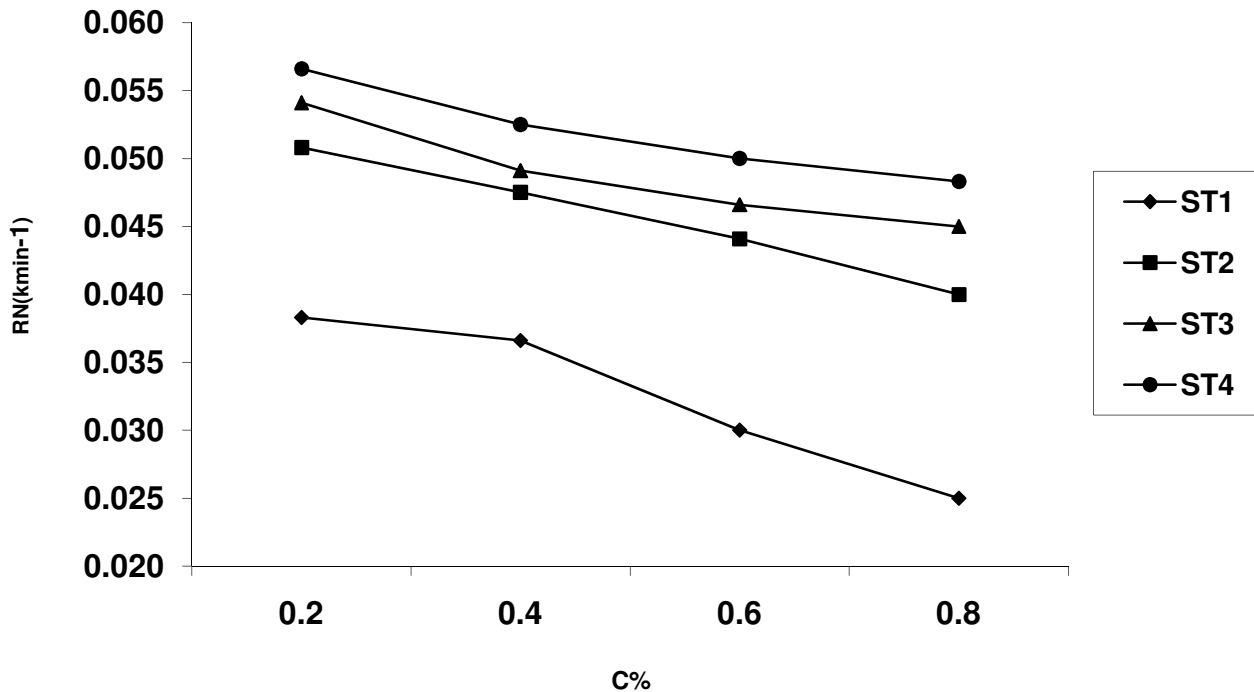


Figure-3
Variation of Reaction Number (RN) with inhibitor concentration (C) for aluminium in 4 N HCl

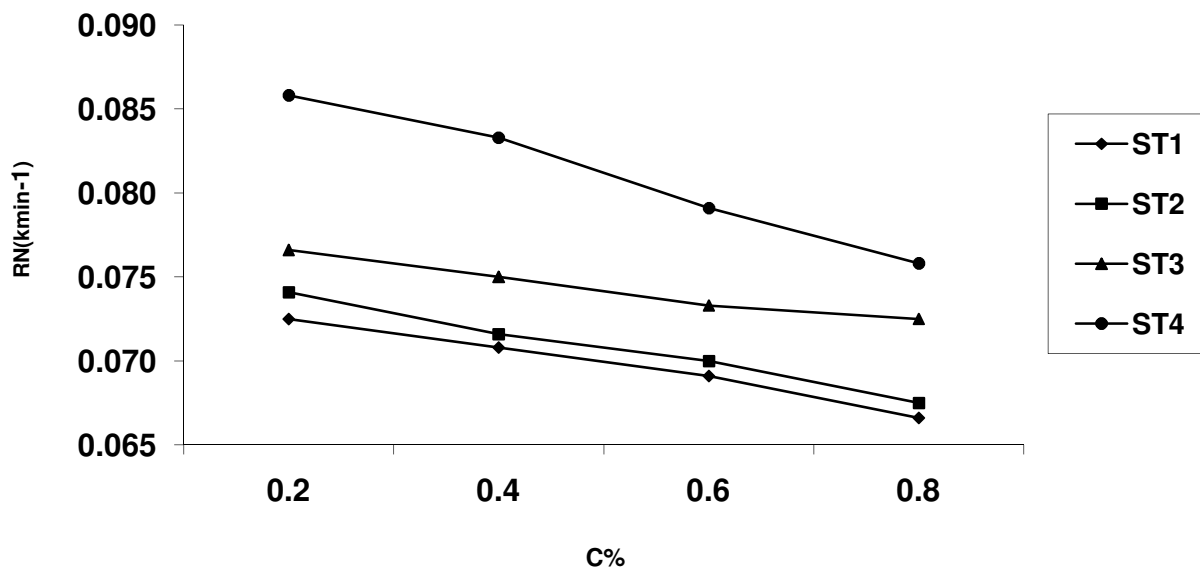


Figure-4
Variation of Reaction Number (RN) with inhibitor concentration (C) for aluminium in 4 N H₂SO₄
ST₁ : 1 - (2, 6-diazene) - 3 -benzyl thiourea, ST₂ : 1 - (3'-pyridyl) - 3 - benzyl thiourea, T₃ : 1 - (3' - pyridyl) - 1 -phenyl thiourea, T₄ : 1 - (2' - pyridyl) - 3 -phenyl thiourea

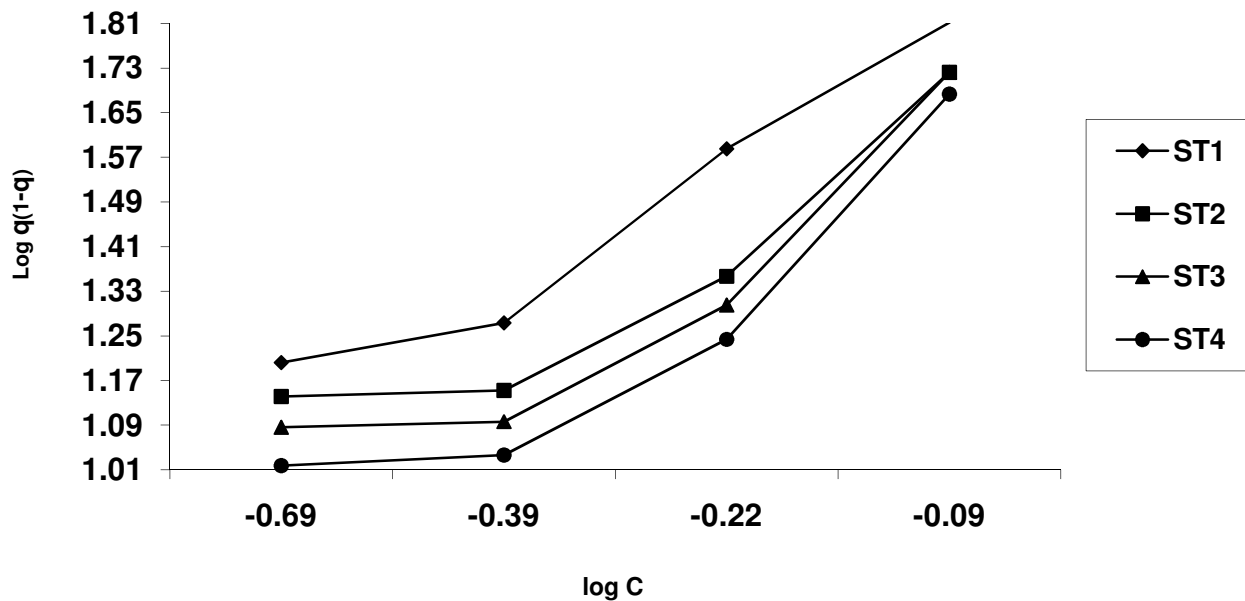


Figure-5
 Langmuir adsorption isotherms for mild steel in 2.5N HCl with inhibitor addition

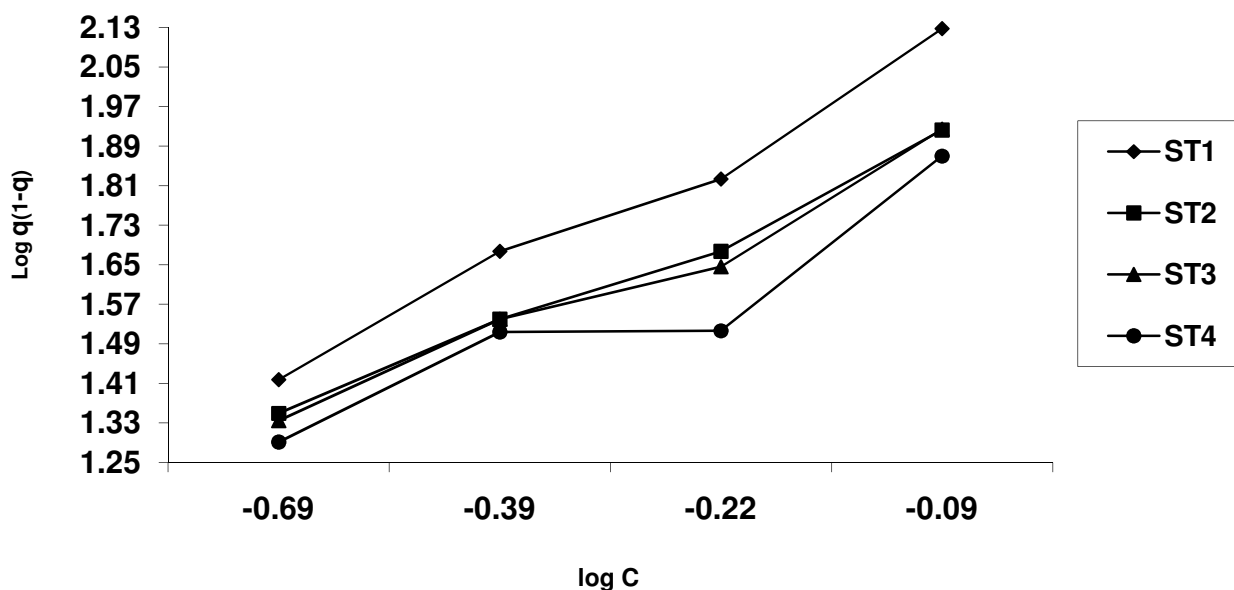
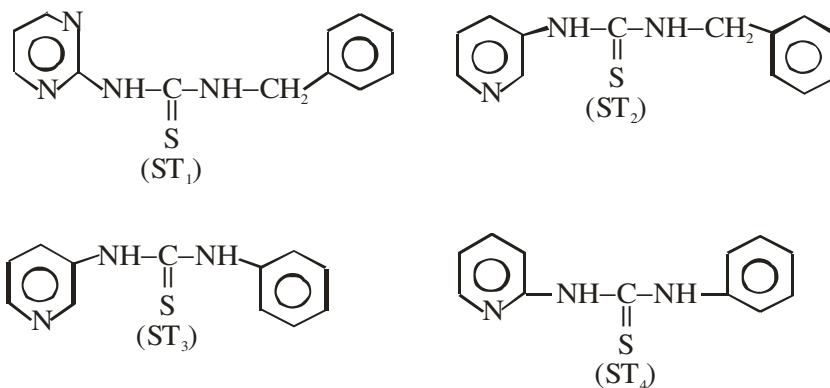


Figure-6
 Langmuir adsorption isotherms for mild steel in 2.5N H₂SO₄ with inhibitor additions
 ST₁ : 1-(2, 6-diazeno) – 3 –benzyl thiourea, ST₂ : 1 - (3' pyridyl) – 3 – benzyl thiourea, ST₃ : 1 – (3' - pyridyl) – 1 –phenyl thiourea, ST₄ : 1 – (2' - pyridyl) – 3 –phenyl thiourea



The results revealed that thiourea compounds effectively reduce the corrosion rates of mild steel in acid solution. The following order of inhibition efficiency has been observed for the four substituted thiourea for Mild Steel $ST_4 < ST_3 < ST_2 < ST_1$.

Organic compounds adsorb on the metal surface forming a barrier between the metal and the corrosive environment. Some structural features of the organic compounds help them to do so. The lone pair electrons of the mentioned atoms facilitate the adsorption process. ST₁ is most effective inhibitor since lone pair of electron present on nitrogen atoms of pyrimidine ring and thiourea moiety is maximum. ST₂ has pyridyl ring having less electron density in comparison to ST₁ hence it is less basic than ST₁. In case ST₃ and ST₄ lone pair present on nitrogen atom attached to benzene take part in the resonance. Hence they are less available for bonding with metal surface.

The substituted thiourea possess N and S as hetero atom and thereby offering an electron rich reaction centre. It has been reported that inhibitors are adsorbed on corroding metal surface through electron rich N and S atom thus forming a chemisorbed monolayer of the inhibitor which acts as a physical barrier between the metal and corrosive solution.

The higher ionization of substituted thiourea in H₂SO₄ than in HCl may be the reason of these compounds exhibiting higher inhibition efficiencies in H₂SO₄ as compared in HCl since increased ionisation of inhibitor molecule will facilitate the adsorption of inhibitor on the Mild Steel surface.

The inhibition efficiency is thus, directly proportional to the fraction of the surface covered with adsorbed inhibitors.

Conclusion

A study of pyridyl substituted thiourea compounds (ST₁, ST₂, ST₃, ST₄) are efficient inhibitors of corrosion of mild steel in sulphuric acid and hydrochloric acid. Both mass loss and thermometric methods have shown that the inhibition efficiency of four inhibitors increases with increasing in

concentration of inhibitor and increases as increasing the concentration of HCl and H₂SO₄. The highest inhibitor efficiency upto 98.48% in 2.5 N HCl and 99.26% in 2.5 N H₂SO₄.

The corrosion rate of mild steel is maximum in HCl. The corrosion rate is governed by number of complex reactions taking place and also the nature of the protective film. Finally it may be concluded from the results that the newly synthesised pyridyl substituted thiourea are efficient corrosion inhibitors for mild steel in H₂SO₄ and HCl.

The inhibition of the compound is governed by chemisorption mechanism in both the acid media. As the molecule approaches the electrode surface the electric field of double layer increases the polarization of molecules and induces additional charges on S and N atom and thus enhances the adsorption of molecules.

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