Triazines: Efficient Corrosion Inhibitors for Mild Steel in Hydrochloric Acid Solution

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Five triazines namely Hexahydro-1,3,5-triphenyl-s-triazine (Inh-1), Hexahydro-1,3,5-p-tolyl-s-triazine (Inh-2), Hexahydro-1,3,5-p-methoxyphenyl-s-triazine (Inh-3), Hexahydro-1,3,5-p-aminophenyl-s-triazine (Inh-4) and Hexahydro-1,3,5-p-nitrophenyl-s-triazine (Inh-5) were synthesized and investigated as corrosion inhibitors of mild steel in 1N HCl solution using weight loss, polarization resistance, Tafel polarization and electrochemical Impedance spectroscopy techniques. The inhibition efficiency of the synthesized inhibitors followed the order Inh4 > Inh3 > Inh2 > Inh1 > Inh5. The inhibiting action of triazines was found to depend on electronic nature of functional groups present in triazines. Potentiodynamic polarizations suggest that triazines are mixed type in nature. Electrochemical Impedance spectroscopy was also used to investigate the mechanism of the inhibition. Thermodynamic parameters were also calculated to know the mechanism of the inhibition. AFM is used to know the surface roughness of the mild steel sample with and without inhibitors.

Keywords: Mild steel; Corrosion inhibition; Polarization; EIS; AFM

1. INTRODUCTION

Acids are widely used in industries such as pickling, cleaning, descaling etc. Inhibitors are very effective to reduce the rate of dissolution of metals. Compounds containing nitrogen, sulphur and oxygen have been reported as inhibitors [1-7]. Organic inhibitors generally protect the metal from corrosion by forming a film on the metal surface. Effectiveness of the inhibition is dependent to the chemical composition, their molecular structure and their affinities for the metal surface. These materials develop a protective film of adsorbed molecules on the metal surface, which provides a barrier to the dissolution of metal in electrolyte solution. The most efficient organic inhibitors are

organic compounds having π -bonds in their structures. The adsorption of these compounds are influenced by the electronic structure of inhibiting molecules, steric factor, aromaticity and electron density at donor site, presence of functional group such as –CHO, –N=N, R–OH etc., molecular area and molecular weight of the inhibitor molecule [8-13].

The primary step in the action of inhibitors in acid solution is adsorption onto the metal surface, which is usually oxide-free. The adsorbed inhibitor then acts to retard the cathodic and / or anodic electrochemical corrosion reaction. It is often not possible to assign a single general mechanism of action to an inhibitor because the mechanism may change with experimental conditions. Thus, the mechanism of inhibition of an inhibitor may vary with factors such as concentration, pH, nature of the anion of the acid and nature of the metal. The action mechanism of inhibitors with the same functional group may additionally vary with factors such as the effect of the molecular structure on the electron density of the functional group and the size of the aromatic and aliphatic protons of the molecule [14-16]. The choice of an appropriate organic inhibitor for a particular system is unfortunately limited due to the variety of corrosion systems and specificity of the inhibitor compounds.

The objective of this study is to investigate the corrosion behaviour of mild steel in 1N HCl solution in the presence of Hexahydro-1,3,5-triphenyl-s-triazine (Inh-1), Hexahydro-1,3,5-p-tolyl-s-triazine (Inh-2), Hexahydro-1,3,5-p-methoxyphenyl-s-triazine (Inh-3), Hexahydro-1,3,5-p-aminophenyl-s-triazine (Inh-4) and Hexahydro-1,3,5-p-nitrophenyl-s-triazine (Inh-5) using weight loss, polarization resistance, Tafel polarization and electrochemical impedance techniques. The effect of different groups on the corrosion inhibition process was also studied. The effects of temperature, acid concentration, immersion time were studied on corrosion inhibition. Several isotherms were tested for their relevance to describe the adsorption behaviour of the compounds studied. AFM study was carried out to investigate the surface morphology of the inhibited and uninhibited mild steel samples.

2. EXPERIMENTAL

2.1. Synthesis of sym-Triazines



If, X= H; Hexahydro-1,3,5-triphenyl-s-triazine (Inh1) X=CH₃; Hexahydro-1,3,5-p-tolyl-s-triazine (Inh2) X= OCH₃; Hexahydro-1,3,5-p-methoxyphenyl-s-triazine (Inh3) X= NH₂; Hexahydro-1,3,5-p-aminophenyl-s-triazine (Inh4) X= NO₂; Hexahydro-1,3,5-p-nitrophenyl-s-triazine (Inh5) The different triazines were synthesized in the laboratory according to the procedure described elsewhere [17]. The synthesized triazines were characterized through its IR and ¹H NMR spectral data and their purity was confirmed by thin layer chromatography. The name and structural formula of the all the triazines are given below:

2.2. Corrosion Study

The mild steel strips having composition (wt %): C 0.14, Mn 0.035, Si 0.17, S 0.025, P 0.03 and balance Fe were used for weight loss as well as electrochemical studies. The test solution of hydrochloric acid (AR grade) is used for all the studies.

2.2.1. Weight loss Studies

Weight loss experiments were conducted on the mild steel strips of $5.0 \times 2.0 \times 0.025$ cm³ size. The study was carried out at 35°C temperatures for 3h time duration in 1N HCl solution in stoppard conical flasks to know the optimum concentration of all the triazines used in the study. The inhibition efficiency (%) and surface coverage (θ) was determined by following equation:

$$I.E.(\%) = \frac{W_o - W_i}{W_o} \times 100$$

$$\theta = \frac{W_o - W_i}{W_o}$$

where, W_o and W_i is the weight loss value in absence and in presence of inhibitor respectively.

The weight loss study was also conducted at temperature range, different immersion time and different concentration of HCl solution at optimum concentration of the various inhibitors.

2.2.2. Electrochemical Studies

The electrochemical studies were carried out in a three electrode cell assembly at 35°C. The working electrode was a mild steel of above composition of 1 cm² area and the rest being covered by commercially available lacquer. A large rectangular platinum foil was used as counter electrode and saturated calomel electrode as reference electrode. The working electrode was polished with different grades of emery papers, washed with water and degreased with acetone. The polarization and impedance studies were carried out using Gamry potentiostat / galvanostat (model G-300) with Gamry EIS software, Gamry Instruments Inc., USA. All the experiments were carried out without purging nitrogen. All the experiments were carried out after stabilizing the OCP.

Polarization resistance studies were carried out form cathodic potential of -0.02 V vs SCE to an anodic potential of +0.02 V vs SCE with respect to open circuit potential at a sweep rate 0.125 mVs^{-1} to study the effect of inhibitor on polarization resistance on mild steel.

The Tafel polarization studies were carried out from cathodic potential of -0.25V *vs*. SCE to an anodic potential of + 0.25V *vs*. SCE with respect to the open circuit potential at a sweep rate of 0.5 mVs⁻¹ without de-aerated to study the effect of inhibitor on mild steel corrosion. The linear Tafel segments of anodic and cathodic curves were extrapolated to corrosion potential to obtain the corrosion current densities (I_{corr}). The corrosion inhibition efficiency (% IE) was evaluated from the measured I_{corr} values using the relationship:

$$I.E.(\%) = \frac{I_{corr}^{o} - I_{corr}}{I_{corr}^{o}} \times 100$$

where, I_{corr}^{o} and I_{corr}^{i} are the corrosion current densities in absence and in presence of various concentrations of the inhibitor.

The impedance studies were carried out using ac signals of 10 mV amplitude for the frequency range from 100 kHz to 0.01 Hz. All the studies were performed after immersion of 30 minutes. The charge transfer resistance values were obtained from the diameter of the semi circles of the Nyquist plots. The inhibition efficiency of the inhibitor was calculated from the charge transfer resistance values using the following equation

$$IE\% = \frac{(1/R_{10}) - (1/R_{11})}{(1/R_{10})} \times 100$$

where, R_{ct}^{o} and R_{ct}^{i} are the charge transfer resistance in absence and in presence of inhibitor. The interfacial double layer capacitance (C_{dl}) values have been estimated from the impedance value using bode plot by the formula

$$\left|Z\right| = \frac{1}{2\pi f_{\max}C_{dl}}$$

3. RESULTS AND DISCUSSION

3.1. Weight loss measurement

It has been found that inhibition efficiency of all these triazines increases with increase in concentration. The maximum inhibition efficiency for each compound was obtained at 300 ppm and further increase in concentration did not cause any appreciable change in the performance of inhibitors.

The effect of immersion time on the inhibition efficiency is shown in Figure 1 (a). All the triazines except Inh5 did not change significantly with immersion time. The inhibition efficiency of Inh5 decreases after 3 hours of immersion time suggesting that it is a weak inhibitor as compared to other inhibitors.

The variation of inhibition efficiency with increase in acid concentration from 0.5 to 2.0 N is shown in Figure 1 (b). It is clear that change in acid concentration from 0.5 N to 2.0 N did not cause

any significant change in inhibition efficiency values of Inh1 to Inh4 this shows that these inhibitors are very effective inhibitor in this range. The inhibition efficiency of Inh5 decreased significantly on increasing acid concentration from 0.5 to 2.0 N.



Figure 1. Variation of inhibition efficiency with (a) immersion time, (b) acid conc. and (c) temperature of solution

The influence of solution temperature on inhibition efficiency is shown in Figure 1 (c). It is observed that inhibition efficiency does not change significantly with increase in temperature from 35 to 65°C. The results of this study show that all the triazines are effective corrosion inhibitors up to 65°C.

3.2. Polarization Resistance

The polarization resistance values of mild steel in 1N HCl with different concentrations (50-300 ppm) of Inh1 to Inh5 are given in Table 1. The R_p values of different triazines at 300ppm concentration are 190.6 ohm (Inh1), 220.9 (Inh2), 277 ohm (Inh3), 476.9 (Inh4) and 92.03 (Inh5). The increase in the R_p values suggests that inhibition efficiency increases with the increase in the inhibitor concentrations. All triazines are effective inhibitors at 300 ppm and they inhibit corrosion by blocking the active sites of metal.

Name of Inhibitor	Inhibitor Concentration	R _p	IE
	(ppm)	(Ω)	(%)
Blank	-	13.99	-
Inh1	50	91.4	84.69
	100	115.2	87.86
	200	147.7	90.52
	300	190.6	92.66
Inh2	50	97.7	85.86
	100	149.2	90.62
	200	189.5	92.61
	300	220.9	93.67
Inh3	50	138.1	89.86
	100	186.5	92.49
	200	211.4	93.38
	300	277.0	94.95
Inh4	50	157.1	91.09
	100	233.2	94.00
	200	361.3	96.13
	300	476.9	97.07
Inh5	50	32.1	56.40
	100	42.2	66.86
	200	63.6	77.99
	300	92.0	84.80

Table 1. Polarization parameters for the corrosion of mild steel in 1N HCl in absence and presence different concentration of inhibitor.

3.3. Tafel Polarization

Figure 2 (a-e) represents the potentiodynamic polarization curves of mild steel in 1N HCl in the absence and presence of various concentrations of the five triazines under study. All these five figures were use to calculate the corrosion potential (E_{corr}), anodic and cathodic Tafel slopes (β_a and β_c), and corrosion current density (I_{corr} .). Table 2 gives the electrochemical parameters i.e. Corrosion potential

(E_{corr}), anodic and cathodic Tafel slopes (β_a and β_c), corrosion current density (I_{corr}), percentage inhibition efficiency (%IE) and corrosion rate (CR). It can be seen from the results that I_{corr} values of all the triazines decrease in the concentration range of 50 to 300 ppm. Maximum reduction of I_{corr} for each triazine is obtained at 300 ppm concentration. It is also observed that β_a and β_c values and E_{corr} values did not change significantly in increase of concentration of triazines, suggesting that all the triazines are mixed type of inhibitors and they inhibits corrosion by blocking the active sites of the metal surface [18].

The inhibition efficiency showed following order Inh4 > Inh3 > Inh2 > Inh1 > Inh5.





Figure 2. Tafel polarization curves of mild steel in absence and presence of (a) Inh1, (b) Inh2, (c) Inh3, (d) Inh4 and (e) Inh5

Name of Inhibitor	Inhibitor concentration	-E _{corr} mV vs SCE	β _a (mV/dec)		I _{corr} (μA/sec)	IE (%)
	(ppm)	1=0				
1N HCl	Blank	470	65.5	107.1	1540.0	-
Inh1	50	473	64.4	161.7	176.0	88.57
	100	478	61.4	147.4	137.0	91.10
	200	480	72.6	185.2	110.0	92.86
	300	478	79.4	181.8	92.1	94.01
Inh2	50	487	61.9	202.7	165.0	89.28
	100	480	69.6	179.8	106.0	93.12
	200	487	65.1	153.1	92.9	93.97
	300	489	77.0	151.8	79.4	94.84
Inh3	50	488	62.1	170.3	135.0	91.23
	100	478	79.4	181.8	92.1	94.02
	200	489	72.1	172.7	84.2	94.53
	300	474	78.4	171.4	68.2	95.57
Inh4	50	480	72.6	185.2	110.0	92.85
	100	490	64.2	141.2	71.6	95.35
	200	479	65.2	150.2	47.3	96.93
	300	486	67.3	128.1	40.1	97.40
Inh5	50	466	67.0	188.5	589.0	61.75
	100	472	66.1	216.4	390.0	74.67
	200	473	60.2	187.8	294.0	80.91
	300	487	61.9	199.5	165.1	89.29

Table 2. Tafel polarization parameters for the corrosion of mild steel in 1N HCl in absence and presence of different concentration (0 to 300 ppm) of inhibitors

3.4. Electrochemical Impedance Spectroscopy

Nyquist representations of different inhibitors are shown in Figure 3a-3e. It is clear from all plots that impedance response of mild steel in test solution was significantly changed after addition of the inhibitors. The several theoretical circuits were applied for the study the impedance behaviour. The standered Randle circuit (Figure 4) was most fit in the present studies that have impedance spectra containing single capacitive semicircle. The circuit composed of uncompensated solution resistance (R_{Ω}), polarization resistance (R_t), double layer capacitance (C_{dl}). Various parameters such as charge transfer resistance (R_{ct}), double layer capacitance (C_{dl}) and percentage inhibition efficiency (%IE) have been calculated and listed in Table 3.

The existence of single semi circle showed the single charge transfer process during dissolution which is unaffected by the presence of inhibitor molecule. Increase of charge transfer resistance and decrease of double layer capacitance with increasing inhibitor concentration indicates that these compounds inhibit the corrosion rate of mild steel by an adsorption mechanism [19]. In fact, the presence of all five compounds increases the value of the charge transfer resistance in the acidic solution, the effect being most pronounced with the Inh4. The increase in charge transfer resistance value is attributed to the formation of protective film on the metal/solution interface [20, 21]. The values of C_{dl} of studied compounds decreased with increasing concentration. The decrease in C_{dl} is attributed to increase in thickness of electronic double layer [22]. The decrease in the values of C_{dl} follows the order similar to that obtained for the I_{corr} studies. This can be explained on the basis of adsorption of triazines on the metal surface [23]. The impedance study also gave the same efficiency trend as found in weight loss, polarization resistance and Tafel polarization methods









Figure 3. Nyquist plot of mild steel in 1N hydrochloric acid with different concentrations of (a) Inh1 (b) Inh2 (c) Inh3 (d) Inh4 (e) Inh5



- Figure 4. Electrical equivalent circuit (R_{Ω} = uncompensated solution resistance, R_t = polarization resistance, C_{dl} = double layer capacitance).
- **Table 3.** Electrochemical impedance parameters for mild steel in 1N HCl in absence and presence different concentrations of inhibitors

Name of Inhibitor	Inhibitor concentration (ppm)	$\frac{R_t}{(\Omega \text{ cm}^2)}$	C _{dl} (μF cm ⁻²)	IE (%)
1N HCl	-	19.8	1555.0	-
Inh1	50	108.3	283.4	81.72
	100	147.3	209.0	86.55
	200	212.0	145.2	90.66
	300	335.5	90.2	94.09
Inh2	50	125.1	246.1	84.17
	100	165.5	186.0	88.03
	200	262.1	117.5	92.44
	300	346.3	88.0	94.28
Inh3	50	160.7	190.6	87.68
	100	293.7	105.0	93.30
	200	349.8	88.1	94.33
	300	440.9	68.5	95.51
Inh4	50	185.5	165.2	89.33
	100	295.6	103.2	93.30
	200	349.5	88.4	94.33
	300	512.8	60.0	96.14
Inh5	50	76.91	400.3	74.26
	100	112.0	275.0	82.32
	200	119.8	256.9	83.47
	300	149.5	206.2	86.56

3.5. Adsorption Isotherm and Thermodynamic parameters

The mechanism of corrosion inhibition may be explained on basis of adsorption behaviour [24]. Several adsorption isotherms were tested to describe the adsorption behaviour of all the compounds used in study. A straight line was obtained on plotting $C_{inh} vs C_{inh} / \theta$ for all the inhibitors

used in study shown in Figure 5. The degree of surface coverage (θ) for different inhibitor concentrations of all inhibitors were evaluated from weight loss data. It is found that all the compounds under study obey Langmuir adsorption isotherm. The plot yields straight lines with slope values are almost unity.

The degree of surface coverage (θ) for different concentrations of inhibitors in 1N HCl at 35-65 °C for 3 h of immersion time has been evaluated from weight loss values. The data were tested graphically by fitting to various isotherms. A plot of log (θ / 1- θ) versus 1/T is given in Figure 6. The value of heat of adsorption was determined from the slope of the graph. The values for heat of adsorption are included in Table 4. Since the values of heat of adsorption for the inhibitors are less that -40 KJmol⁻¹ suggests physical adsorption of the inhibitors on the metal surface [25].



Figure 5. Langmuir adsorption isotherm plot for the adsorption of different triazines in 1N HCl, on the surface of mild steel.



Figure 6. Adsorption isotherm plot for log $(\theta / 1 - \theta)$ versus 1/T

It has been reported by number of authors [26-28] that in acid solution, logarithm of the corrosion rate is a linear function with 1 / T (Arrhenius equation):

$$\log(rate) = \frac{-E_a^\circ}{2.303RT} + A$$

Where, E_a° is the apparent effective activation energy, R general gas constant and A is the Arrhenius pre-exponential factor. A plot of log of corrosion rate obtained by weight loss measurement versus 1/T gave straight line as shown in Figure (7a). The values of activation energy – (E_a°) obtained from the slope of the lines are given in Table 4.





Figure 7. Adsorption isotherm plot of log (CR) versus 1/T; (c) Adsorption isotherm plot for log (CR/T) versus 1/T

An alternative formula of the Arrhenius equation is the transition state equation [29]:

$$Rate = \frac{RT}{Nh} \exp\left(\frac{\Delta S^{\circ}}{R}\right) \exp\left(-\frac{\Delta H^{\circ}}{RT}\right)$$

where, h is plank's constant, N the Avogadro's number, ΔS° the entropy of activation and ΔH° the enthalpy of activation. A plot of log (CR / T) versus 1/T gave a straight line (Figure 7b), with a slope of ($-\Delta H^{\circ}/2.303$ R) and an intercept of [log(R/Nh) + ($\Delta S^{\circ}/2.303$ R)], from which the values of ΔS° and ΔH° were calculated and listed in Table 4. The data shows that thermodynamic activation functions (E_a°) of the corrosion in mild steel in 1N HCl solution in the presence of the inhibitors is lower than those in free acid solution indicating that all the inhibitors exhibits high inhibition efficiency on increasing the temperature [30]. The negative values of ΔS° and ΔH° indicates that the process of adsorption is exothermic, spontaneous [31, 32].

Free energy of adsorption (ΔG_{ads}) calculated using the following equations [33] are given in Table 4.

$$\Delta G_{ads} = -RT \ln(55.5K)$$
$$K = \frac{\Theta}{C(1-\Theta)}$$

where, θ is degree of coverage on the metal surface, C is concentration of inhibitor in mol/L, R is a constant and T is temperature. The ΔG_{ads} value of all the inhibitors are found that less than 40KJmol⁻¹ indicating that all the inhibitors are physically adsorbed on metal surface [34]. The negative values of ΔG_{ads} indicated the spontaneous adsorption of inhibitors on surface of mild steel [35].

Table 4. Thermodynamic parameters for mild steel in 1N HCl in absence and presence of 300 ppm concentration of Inh1-Inh5

Name of Inhibitor	E _a (kJmol ⁻¹)	-ΔH (Jmol ⁻¹ K ⁻¹)	-ΔS (Jmol ⁻¹ K ⁻¹)	-ΔG (kJmol ⁻¹)	-Q (kJmol ⁻¹)
Blank	33.84	31.36	111.41	-	-
Inh1	16.64	14.06	188.94	16.73	19.06
Inh2	23.22	20.65	169.82	17.97	11.15
Inh3	19.43	16.86	187.47	19.78	14.99
Inh4	26.02	23.45	171.25	21.19	13.24
Inh5	28.97	26.40	142.20	15.45	5.96

3.6 Surface Characterization: AFM study

Surface morphology of the polished mild steel and mild steel in 1N HCl in absence and presence of inhibitors were investigated through Atomic Force Microscopy (AFM) technique. The

results were shown in figure (8a-g). The average roughness of polished mild steel (Figure 8a) and mild steel in 1N HCl without inhibitor (Figure 8b) was calculated as 48 nm and 471 nm. It is clearly shown in Figure (8b) that mild steel sample is getting cracks due to the acid attack on mild steel surface. However in presence of optimum concentration of Inh1, Inh2, Inh3, Inh4 and Inh5 (showed in Figure 8c-g) the average roughnesses were reduced to 104 nm, 100 nm, 91 nm, 80 nm and 205 nm. The calculated roughness for the Inh4 is lowest in all the inhibitors also reveals that this inhibitor protects the mild steel surface more than the all other triazines derivatives form the 1N HCl solution.



С



Figure 8. AFM study of the mild steel surfaces with and without inhibitor (a) polished mild steel (b) Mild Steel in 1N HCl (c) Mild steel in 1N HCl with Inh1 (d) Mild steel in 1N HCl with Inh2 (e) Mild steel in 1N HCl with Inh3 (f) Mild steel in 1N HCl with Inh4 (g) Mild steel in 1N HCl with Inh5

4. MECHANISM OF INHIBITION

Corrosion inhibition of mild steel in hydrochloric acid solution by different triazines can be explained on the basis of molecular adsorption. The compounds inhibit corrosion by blocking both anodic and cathodic sites. In acidic solutions the triazines exist as protonated species. These protonated species are adsorbed on the cathodic sites of the mild steel and decrease the evolution of hydrogen. The adsorption on anodic site occurs through π electrons of aromatic rings and lone pair of electrons of different hetero atoms which decrease the anodic dissolution of mild steel [36]. The high performance of symmetrical triazines is attributed to the presence of π electrons, quaternary nitrogen atoms, larger molecular size and the planarity of these compounds. In the present study the order of inhibition efficiency is found as follows:

Inh4>Inh3>Inh2>Inh1>Inh5

The highest inhibition efficiency of the Inh4 is attributed to presence of highly electron donating $-NH_2$ group. Inh5 showed lowest inhibition efficiency among the studied compounds due to presence of electron withdrawing NO₂ group. The better performance of Inh3 and Inh2 than Inh1 is attributed to +R and +I effect of OCH₃ and CH₃ groups respectively which increase electron density at triazines thereby favouring stronger adsorption of Inh3 and Inh2 on the metal surface than Inh1.

5. CONCLUSIONS

All the triazines showed highest inhibition efficiency at 300 ppm concentration. The trend of inhibition efficiencies of all triazines at optimum concentration are as follows:

Inh4>Inh3>Inh2>Inh1>Inh5

Presence of NH_2 group significantly increased the inhibition efficiency of Inh4 while withdrawing group NO_2 reduced the inhibition efficiency of Inh5. All the triazines were found to obey Langmuir adsorption isotherm. All the triazines were found to act as mixed inhibitors.

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