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Corrosion Inhibition of Brass in 0.5N HNO_3 By 3-Hydroxy-3-(4-Chlorophenyl-1-(4-Sulphonato (Sodium Salt)) Phenyl Triazene (HCST): Adsorption and Thermodynamic Study

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ABSTRACT

The effect of the concentration and nature of novel 3-hydroxy-3-(4-chlorophenyl-1-(4-sulphonato (sodium salt)) phenyl triazene (HCST) on corrosion and dezinfication of 70/30 brass in 0.5N HNO_3 *solution has been investigated by weight loss method. Inhibition efficiency of this corrosion inhibitor was also evaluated at different temperature ranging from 303K-333K. It was observed that HCST showed 88.13% inhibition efficiency at 303K upto 0.005M concentration of inhibitor. Weight loss method has been used to analyze the corrosion behavior of the brass in the absence and presence of different concentration of inhibitor. The corrosion inhibition efficiency increases with increasing concentration of inhibitor and decreases with rise in temperatures. Activation energy (*E_a*), Enthalpy (*ΔH*), Entropy (*ΔS*) and Gibbs free energy (*ΔG*) for corrosion process have also been calculated. The adsorption behavior of HCST on brass surface has been found to obey Langmuir adsorption isotherm. Results reveal that hydroxytriazenes can be potential corrosion inhibitors.*

Keywords: Hydroxytriazene (HCST), Corrosion inhibitors, Weight loss method, Brass, Nitric acid.

INTRODUCTION

Dezincification of brass is one of the well-known and common processes by means of which brass loses its valuable physical and mechanical properties leading to structural failure [1]. Corrosion having two specific forms are direct chemical attack and electrochemical attack and in this both types of corrosion, the metal is converted into a metallic compound such as an oxide, hydroxide, or sulfate. Brass has been the most commonly used material for fittings such as stop valves and control valves, shipboard condensers, power plant condensers and petrochemical heat exchangers [2-4]. Due to various industrial applications and economic importance of brass, its protection against corrosion attracts much attention .One of the most important methods in corrosion protection is to use inhibitors [5-8].

Many organic compounds, especially those containing polar groups and/or substituted heterocycle including nitrogen, sulphur and oxygen in their structures, have been reported to inhibit the corrosion of copper [9-12]. The inhibiting action of these organic compounds is usually attributed to the formation of donor–acceptor surface complexes between the free or π electrons of an inhibitor and the vacant d-orbital of a metal and adsorption [13-14].

Hydroxytriazenes have been extensively used as spectrophotometric and complexometric reagents for estimation of almost transition metal series. A number of voluminous reviews have been published by our laboratory on application of hydroxytriazenes [15-21]. Not only this, they have also been screened for their biological activities as evidenced by papers appearing in the literature. However, very few studies have been done on their corrosion inhibitive properties. The studies have been done on their behavior for protection of brass in HNO_3 medium. It is proven that hydroxytriazenes are potential corrosion inhibitors for brass and copper in ammonical medium. This paper focuses on application of hydroxytriazenes for their corrosion inhibition.

MATERIALS AND METHODS

Synthesis of 3-hydroxy-3-(4-chlorophenyl-1-(4-sulphonato (sodium salt)) phenyl triazene

Step I-Reduction of nitro compounds: In this step 0.05 mol of p-chloronitrobenzene were reduced with Zn dust in the presence of NH_4Cl in water-alcohol medium using mechanical stirrer at 40-60°C to obtain phenyl hydroxylamine. The resulting mixture was filtered under suction and the filtrate was kept in fridge to cool.

Step II- Diazotization of sulphanilic acid: In this step 0.05 mol sulphanilic acid was dissolved in solution of sodium carbonate and cooled to 0-5°C. Sodium nitrite solution was added to sulphanilic acid solution in acidic medium at 0-5°C under constant mechanical stirring to obtain p-sulphonatophenyldiazonium chloride.

Step III- Coupling: The diazonium compound prepared in step II was added slowly to the phenyl hydroxylamine (product of step I) solution under constant stirring at 0-5°C and pH 5-6 was maintained by adding solid sodium acetate. The reaction mixture was further stirred for 15 min after complete addition of diazonium compound. Sodium chloride was added in sufficient quantity to saturate the solution. Final product obtained was soluble in water so washing with H_2O was avoided. The crude compound was recrystalized from double distilled water. Purity of Compounds were checked by recording melting points and other physical characterization methods such as IR, elemental analysis,^{1} H NMR and Mass. It was found that the compounds synthesized are in pure state.

Characterization: IR in cm^{-1}: 3463 (O-H str.), 3207(N-H str.), 3050 (C-H str. Ar), 2948 (C-H str., CH_3), 1632 (N=N str.), 1419 (N-Nstr.), 1320 (N-O str) ,737(C-Cl str.Ar.).The spectra showed the compound to be in pure state. IR spectra were recorded on FT IR. NMR-12.17 δ (ppm) for (O-H, s), 7.47-8.11 δ for multiplet, 8H.

Specimen preparation: The brass specimen having the chemical composition of Cu ~70% and Zn~ 30% were used for experiments with the dimensions (2.5x2.5x0.1) cm. Each specimen was polished with emery paper and rinsed by distilled water, washed with acetone and dried in oven and then accurate weight of specimens were determined using an analytical balance of 0.0001g accuracy. These specimens were placed in beakers having 100 mL solution of 0.5N nitric acid with and without inhibitor using 'V' shape hanging capillary.

Weight loss method: After initial weighing, the two specimens were immersed in 100 mL of nitric acid solution in the presence and absence of different concentration of inhibitor at various temperatures ranging from 303-333K. Thermostated water bath was used to maintained temperature during 24 h immersion ,the specimens were taken out, washed, dried and final weight were noted. From initial and final weight of brass specimen, weight loss in gm, corrosion rate (CR) in g cm^{-2} h^{-1}, percentage inhibition efficacy (IE) and surface coverage (θ) were determined using equation (1, 2, 3) respectively [22-24].

$$CR = \frac{\Delta W}{At} \qquad (1)$$

Where CR is corrosion rate (g cm^{-2} h^{-1}), ΔW is the weight loss (g), A the surface area and t is the immersion time in h.

$$IE\% = \frac{W_o - W}{W_o} \text{ X } 100 \qquad (2)$$

$$\theta = \frac{W_o - W}{W_o} \qquad (3)$$

Where W_o is weight-loss in absence of the inhibitor and W is weight-loss in presence of the inhibitor.

Atomic adsorption spectroscopy: This spectroscopy has been used to find out concentration of dissolve copper and zinc in medium solution in which metal / alloy get corroded in absence of inhibitor and presence of inhibitor. The concentrations of dissolved copper and zinc in mg L^{-1} in blank solutions and solution containing $5x10^{-3}$ mol of the HCST after corrosion at 303K are shown in table 4 and Percent IE have been calculated using following equation (4) and given in table 4.

$$\% \text{ IE} = C_o - C_e / C_o \qquad (4)$$

Where C_o is the concentration of Cu and Zn in without inhibitor solution (Blank), C_e is the concentration of Cu and Zn in with inhibitor solution**.**

RESULTS AND DISCUSSION

Effect of concentration of HCST on corrosion rate and inhibition efficiency: The effect of concentration on corrosion rate of brass in the absence and presence of various concentrations of inhibitor (HCST) were studied at 303 to 333K respectively in 0.5N nitric acid medium. The result (Fig.1) shows that the corrosion rate of brass decreases with increasing the concentration of the inhibitor at all temperature in 0.5N HNO_3 medium which shows an increase in adsorptions of the inhibitor on the surface of the brass. The inhibition efficiency of corrosion inhibitor for brass increases as the concentration of inhibitor increases and decreases with the increasing temperature. The % IE of HCST is due to the adsorption of its molecule on the brass metal surface. This also depended on number of active site in the molecule, molecular size and mode of interaction between metal and inhibitor.

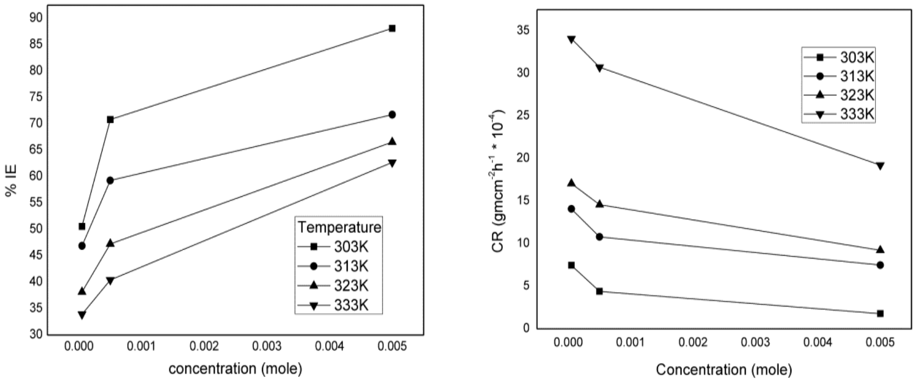

Fig 1: Effect of concentration on IE and Corrosion rate.

Table 1: Dependence of corrosion rates on concentration of HCST at various temperatures

Inhibitors	**Inhibitors concentration (Mole)**	**303K**		**313K**		**323K**		**333K**	
		CR x10^{-4}	**%IE**	**CR x10^{-4}**	**%IE**	**CR x10^{-4}**	**%IE**	**CR x10^{-4}**	**%IE**
Without inhibitor	**Blank**	15.11		26.58		27.64		51.64	
HCST	**5x10^{-5}**	7.46	50.59	14.10	46.92	17.08	38.18	34.10	33.95
	5x10^{-4}	4.40	70.84	10.81	59.31	14.59	47.29	30.74	40.45
	5x10^{-3}	1.79	88.13	7.5	71.78	9.24	66.57	19.24	62.74

Effect of temperature on inhibition efficiency and corrosion rate: The temperature dependence of IE has been worked out by plotting graph between temperature and % Inhibition efficiency (IE). The corrosion rate of brass in nitric acid solutions were studied with and without inhibitors (HCST) at different temperatures ranging from 303 to 333 K. The resultant observations are shown in table 1. Fig. 2 is typical plot of HCST showing that as the temperature increases the inhibition efficiency decreases. The maximum percentage inhibition is 88.13% at the highest concentration 0.005M at 303K.

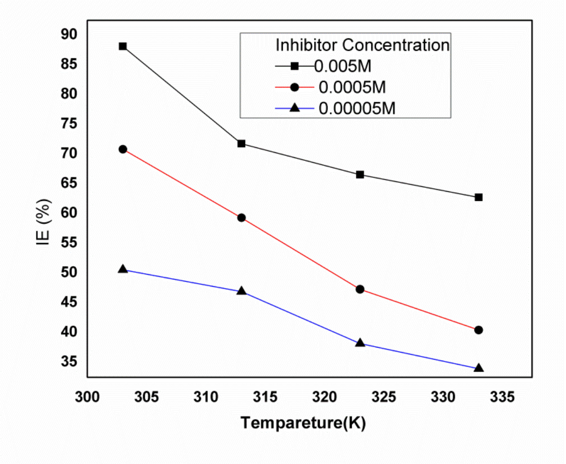

Fig 2 The temperature dependence of % IE

Adsorption isotherm: Adsorption isotherms are used in determining the mechanism of any organo electrochemical reaction. Inhibition Efficiency (%) is directly proportional to the surface covered by the adsorbed molecule (θ). The adsorption isotherm shows relationship between the surface coverage with the adsorbed species and the concentration of inhibitor in solution. The value of the surface coverage (θ) at different concentrations of the inhibitors in 0.5N HNO_3 acid solution was fit to adsorption isotherms. HCST inhibitor is found to obey Langmuir adsorption isotherm because the plot of C/θ vs. C is linear. The isotherm is described by the equation (5) [25, 26].

$$C/\theta = 1/K + C \qquad (5)$$

Where C is the Inhibitor concentration, K is the Adsorption equilibrium constant and θ is the Surface coverage.

Thermodynamic parameters

Determination of Activation energy: The temperature dependence of inhibition efficiency and activation energy in the presence and absence of inhibitor gives mechanism of inhibitor adsorption. Inhibition efficiency decreases with increasing temperature and activation energy (E_a) increases in the presence of

inhibitor compared to absence of inhibitor [27]. Thus the activation energy (E_a) for the corrosion process with and without of the corrosion inhibitor was evaluated from Arrhenius equation (6).
The mathematical expression between the corrosion rate (CR) and temperature (T) is generally expressed by the Arrhenius equation [28, 29].

$$\log CR == \frac{-Ea}{2.303R} + \log A \qquad (6)$$

Where CR is the rate of corrosion, A is the frequency factor, E_a is the apparent activation energy, R is the molar gas constant and T is the absolute temperature.

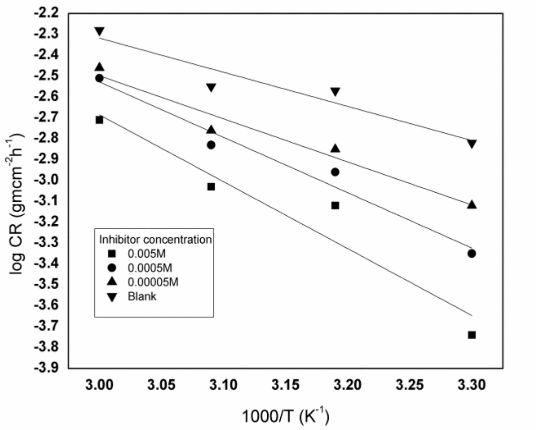

Fig 3. The Arrhenius plot of log CR vs.1/T in presence and absence of inhibitor (HCST)

Fig 3 Curve shows the Arrhenius plot in absence of inhibitor and presence of inhibitor in solutions. Regression coefficients were found very close to 1 which shows a good relationship between log CR and 1/T. Straight lines were obtained with slope of $-E_a/2.303R$. The activation energy (E_a) were calculated from the slope of Arrhenius plot (E_a = 2.303 x 8.314 x slope). The activation energy increase as the concentration of inhibitors (HCST) increases which indicated physical adsorption on the brass surface. The activation energy of corrosion process was greater than 20 kJ mol^{-1} [30] .The values of E_a for the corrosion reaction was calculated and has been given in table 2.

The values of activation energy ($E_{a)}$ for Brass corrosion in the presence of HCST in 0.5N HNO_3 are shown in the table 2. The data show that the activation energy (E_a) in the presence of HCST is higher than without inhibitor. Activation energy E_a was found to be 31.28KJ mol^{-1} for 0.5N HNO_3 and increased to 61.31 KJ mol^{-1} in presence of 0.005N concentration of inhibitor at 303K, showing that the adsorbed organic molecule provides a physical barrier to the charge and mass transfer, leading to reduction in corrosion rate [31].

Table 2: Energy of Activation in KJ mol^{-1} at various concentrations

Concentration of inhibitor	Slope	Activation energy (E_a)KJ/mol
Blank	-1.63	31.28
5x 10^{-5}	-2.06	39.48
5x 10^{-4}	-2.65	50.75
5x 10^{-3}	-3.20	61.31

Determination of Enthalpy and Entropy: Other thermodynamic parameters such as enthalpy (ΔH) and entropy (ΔS) of activation of corrosion process may be evaluated from the effect of temperature. The enthalpy (ΔH) and entropy (ΔS) of activation of corrosion process was calculated from the equation (7) (Transition state theory) [32].

$$CR= \frac{RT}{Nh}\exp\left(\frac{\Delta S}{R}\right)\exp\left(\frac{-\Delta H}{RT}\right) \qquad (7)$$

Where CR is the corrosion rate ($g.cm^{-2}.h^{-1}$), h is the Plank's constant (6.62×10^{-34} J.s), N is the Avogadro's number (6.022×10^{-23} mol^{-1}), R is the universal gas constant (8.3144 $J.K^{-1}.mol^{-1}$), and T is the absolute temperature.

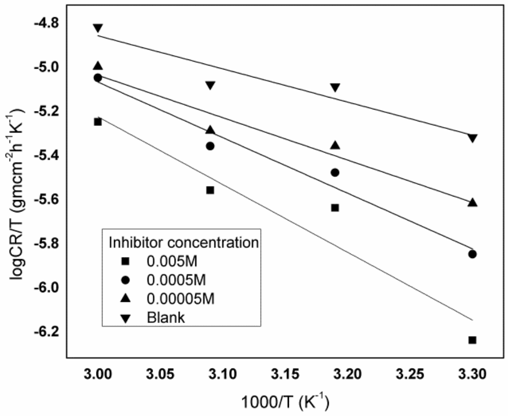

Fig:4 Plot of log (CR/T) and (1000/T) for Brass in uninhibited and inhibited 0.5N HNO_3 solution

Fig. 4 is a plot of log CR vs1000/T is a straight line graph with a slope of ($-\Delta H/2.303R$) and an intercept of ($\log R/Nh + \Delta S/2.303R$) from which the values of ΔH (ΔH = Slope × R) and ΔS (ΔS= (Intercept- lnR/Nh) 2.303R) were calculated [33, 34]. The results presented in Table 3 shows that the enthalpy of activation values are positive which reflects the endothermic nature of metals or brass dissolution process [35]. Also, the entropies of activation energy were positive indicating that the activation complex represents association steps and that the reaction was spontaneous and feasible [36].

Table 3 Enthalpy and Entropy of the reaction with various concentrations of HCSTC

Inhibitors name	Conc. of inhibitors	ΔH (kJ/mol)	ΔS (J/mol K)
3-Hydroxy-3-(4-chlorophenyl-1-(4-sulphonato(sodium salt)phenyltriazene	Blank	28.78	-203.59
	$5x10^{-5}$	36.80	-182.96
	$5x10^{-4}$	48.25	-149.22
	$5x10^{-3}$	58.81	-120.55

Determination of Gibbs Free energy: The Gibbs free energy of adsorption(ΔG) characterize the interaction of adsorption molecules and metal surface, which was calculated by equation (8). The values of ΔG obtained are presented in Table 4. The negative values of ΔG ensure the spontaneity of adsorption process and stability of the adsorbed layer on the brass surface. Generally, the values of ΔG around -20 kJ/mol or lower are consistent with physiosorption, while those around -40kJ/mol or higher involve chemisorptions. As shown in the Table 4, results obtained indicate that the values of ΔG are negative. This implies that the HCST is adsorbed on the surface of Brass specimen and so gives a very strong inhibition of corrosion. The values of ΔG were calculated by the slope of plot of C/θ vs. C which shows langmuir adsorption isotherm. Compound HCST which used as an inhibitor is found to obey Langmuir adsorption isotherm because the plot of C/θ vs C is linear.

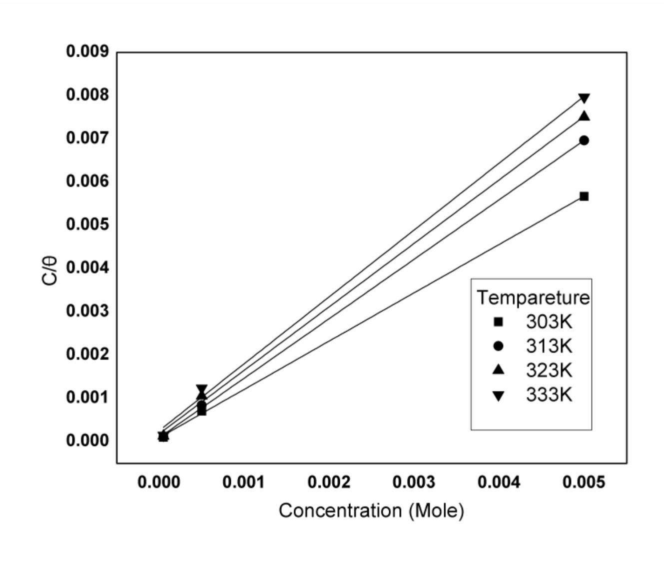

Fig 5. The plot of (C/θ) vs. C was linear with the intercept equal to 1/K for the HCST.

The values for Slope and correlation coefficient (r^2) for the HCST at various temperatures are shown in the Table 4.

$$\Delta G = -2.303RT \log (55.5K) \qquad (8)$$

Table 4: Gibbs free energy at various temperature of 3-Hydroxy-3- (4-chlorophenyl-1-(4- sulphonato (sodium salt)) phenyl triazene (HCST)

Temperature(K)	Slope	ΔG(kJ/mol)	R^2
303	1.11	-33.51	0.99941
313	1.33	-34.58	0.99951
323	1.46	-33.88	0.9978
333	1.54	-34.00	0.99567

Atomic adsorption spectroscopy- In this corrosion studies the concentration of corroded and protected Cu and Zn were determined by AAS and the result have been shown in Table 5. The results indicate that out of brass sample Zn is corroded at higher rate than Cu. Further, the inhibitors are more effective to protect corrosion in Case of Zn more than copper. And the % IE by this HCST is 93.29%., We can explore this corrosion inhibitor for brass and zinc.

Table 5. Concentrations of dissolved Cu and Zn in solution of 0.005M HCST at 303K.

Inhibitors	**Cu Concentration(ppm)**	**%IE**	**Zn Concentration(ppm)**	**%IE**
Blank(without inhibitor)	38.85		399.5	
HCST	29.6	23.80	26.65	93.32

APPLICATIONS

The corrosion behavior of hydrotriazene on brass in nitric acid medium has been investigated using weight loss method. This compound is very effective and low cost. This is applicable as an acid corrosion inhibitor in the industrial field as components in dezinfication.

CONCLUSIONS

The main conclusions are as follows-

1. 3-Hydroxy-3- (4-chlorophenyl-1-(4-sulphonato (sodium salt) phenyl triazene (HCST) shows strong inhibitive action against the corrosion of brass in 0.5N HNO_3.
2. The inhibition efficiency increases with increasing inhibitor concentration upto 0.005M and decreases as the temperature increases.
3. The adsorption of hydroxytriazene on brass obeys Langmuir adsorption isotherm.
4. Hydroxytriazenes are better corrosion inhibitors due to the π-electrons contribution of benzene ring and presence of more adsorption sites. And this significant are introducing phenyl group and SO_3 H group in compounds.

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