# Thermodynamic, Kinetics, Mechanical, Electrical and Surface Study of Corrosion Inhibitor for Mild Steel in Acidic Environment

**V. SAINI[1], S. KUMAR[2], H. KUMAR[3], B.KUMAR[4]**

[1,4]Dept. of Chemistry, Janta Girls P.G. College, Ellenabad, Haryana – 125102, India.
[2]Dept. of Physics, M.D. College Rawatsar, Rajasthan – 335524, India.
[3]Dept. of Chemistry, Ch. Devi Lal University, Sirsa, Haryana – 125055, India.
Email: [1]dony84@rediffmail.com

## Abstract

*Thermodynamic, kinetic, mechanical, electrical and surface study of mild steel in 1.0 M HCl solution with dithizone (DTZ) as corrosion inhibitor were performed by using gravimetric analysis, adsorption isotherm analysis, resistance gradient analysis and analysis of different elastic constants like Young's modulus (Y), Bulk modulus (K) , Shear modulus (η) and Poisson's ratio (σ). DTZ is very effective corrosion inhibitor at atmospheric conditions and high temperatures also, in HCl solution due to its basic nature and adsorption action on mild steel surface by many lone pair sites and unsaturated π delocalized system present in its structure. The rate of increase in resistance gradient due to corrosion action of mild steel decreases significantly with the increased concentrations of inhibitor and increases negligibly with increase in temperature, these results prove the retardation of loss in mechanical strength and surface erosion due to inhibition action of DTZ. Increased value of Poisson's ratio suggests the reduction in longitudinal strain which checks the loss of hardness and strength of mild steel by action of strong acidic environment. Effect of temperature was non significant in acidic solution with inhibitor. Type of adsorption (Langmuir Adsorption isotherm) and mechanism of the inhibition action of DTZ are also reported in this study.*

**Keywords:** *Elastic constants, Langmuir isotherm, Poisson's ratio, Resistance gradient.*

## Introduction

Corrosion has received considerable attention because of the staggering associated costs that result [1-7]. With large number of outdoor structures such as buildings, fences, bridges, towers, automobiles, ships and innumerable other applications exposed to the different environment, there is no wonder that so much attention has been given to the subject. Considerable amount of work has been carried out on corrosion in acidic medium [8-14] however there has been little work concerning the effect of corrosion on the elastic coefficients of metal and the effect of inhibitor on the mechanical and electrical nature of metal. Some reported literature shown that M.Fatmi et. al. [15] worked on structural elastic optical and thermal properties of $Ni_3Al$, H. Liu [16] on mechanical properties and corrosion behavior of novel $Cr_2Ni$ low alloy construction steel with weight loss method, J.H. Hwang et. al. [17] on mechanical properties of FRP bars by hybridizing with steel wire, P. Pokorny et. al. [18] studied the influence of corrosion on zinc powder on mechanical properties of concrete by using compressive cubic strength and compression modulus of elasticity and A. Mukherji et. al. [19] studied the mechanical properties, microstructures and corrosion behavior of Al 7075 and T651 alloy with varying strain rate. In continuation to our earlier study on corrosion inhibition [20-27], in the present study, the corrosion inhibiting properties of DTZ is investigated on mild steel in 0.5 M hydrochloride solution at 30°C by gravimetric analysis, adsorption isotherm analysis, resistance gradient analysis and analysis of different elastic constants like Young's modulus (Y), Bulk modulus (K), Shear modulus (η) and Poisson's ratio (σ).

## Experimental Work

### Materials

Mild steel (ASTM-283) wire (dimensions: 1.58 mm diameter and 60 cm length) of chemical composition: C–0.17, Si–0.35, Mn–0.42, S–0.05, P–0.20, Ni–0.01, Cu–0.01, Cr–0.01 and Fe-balance (w/w) were used. 1.0 M analytical grade HCl solution was used as aggressive acid environment, triply distilled water for solution preparation, Ethyl alcohol and acetone as drying agent, 0.02g/L, 0.04g/L and 0.06g/L DTZ as corrosion inhibitor in HCl solution.

### Equipments

*Weighing balance*- Single pan analytical balance, Precision 0.01mg, Model AB 135-S/FACT, Source Mettler Toledo, Japan. *Air thermostat*- Nine adjustable chambered, Electrically controlled, Accuracy ± 0.1$^o$C. *Maxwell's Needle Apparatus*- Brass tube opened at both ends fitted with four cylinders of equal length and radii (two solid and two hollow), Needle carries a mirror and pin-vice fixed to center, timing measurement by digital stopwatch using telescope. *Searle's Apparatus*- Similar and equal bar of rectangular cross section suspended at middle points from rigid support by two torsion less vertical threads, timing measurement by digital stopwatch using telescope. *Carey Foster Bridge*- A meter long nichrome wire between thick copper strips fixed on wooden board, 1.5 V dry cell, and precision $10^{-4}\Omega$.

### Methods

#### *Weight loss Measurements*

The effects of immersion time and gravimetric experiments were observed on mild steel wire polished successively with sand paper, washed in distilled water, degreased with ethyl alcohol and acetone and finally dried by hot air blower. Tests were conducted in 200 ml acid solutions without and with different concentrations of DTZ inhibitor at 50$^o$C temperature for 36 hours immersion time. The cleaned sample wires were weighed before and after immersion for weight loss measurements. Corrosion rate (CR), degree of surface coverage ($\theta$) and Percentage corrosion inhibition efficiency (PCIE) were calculated using equations 1, 2 and 3 respectively.

$$CR = \frac{534 \times W}{DAT} \tag{1}$$

Where, $W$ is weight loss of wire in mg, $D$ is density of wire in g/cm$^3$, $A$ is surface area of wire in inch$^2$ and $T$ is immersion time of corrosion test in hours.

$$\theta = \frac{W_O - W}{W_O} \tag{2}$$

Where, $W_o$ is weight loss of mild steel wire without inhibitor treatment and $W$ is weight loss of wire with inhibitor treatment.

$$PCIE = \frac{CRo - CR}{CRo} \times 100 \tag{3}$$

Where, $CR_o$ is corrosion rate in absence of inhibitor and *CR* is corrosion rate in presence of inhibitor.

***Mechanical Elastic Coefficient Measurements***

To determine mechanical elastic coefficients, Maxwell's Needle experiment and Searle's experiments were carried out on sample wires corroded in acidic solution, without and with different concentrations of inhibitor. Modulus of rigidity (η) and Young's Modulus were determined by Maxwell's Needle experiment from equation 4 and Searle's experiment from equation 5 respectively.

$$\eta = \frac{8\pi\ell(m_1 - m_2)a^2}{(t_1^2 - t_2^2)r^4} \tag{4}$$

Where, *l* is length and *r* is radius of suspended sample wire, $m_1$ and $m_2$ are mass of each of solid cylinder and hollow cylinder respectively, $t_1$ is time period of oscillation when solid cylinder outside and $t_2$ is time period of oscillation when solid cylinder is inside.

$$\mathrm{Y} = \frac{8\pi\ell I_1}{t_1^2 r^4} \tag{5}$$

Where, *l* is length and r is radius of suspended sample wire, $I_1$ is found from dimensions and mass of bar of Searle's apparatus and $t_1$ is time period of oscillations. Having determined *η* and Y, the value of bulk modulus (K) and Poisson's ratio (σ) were calculated using equations 6 and 7 respectively.

$$\frac{1}{\mathrm{Y}} = \frac{1}{9K} + \frac{3}{\eta} \tag{6}$$

and

$$\sigma = \frac{1}{2}\left(1 - \frac{\mathrm{Y}}{3K}\right) \tag{7}$$

***Resistance Gradient Measurements***

To determine the resistance gradient of sample wires using Carey Foster Bridge, first they were cut in equal lengths and then tested for resistance, using the formula

$$\rho = \frac{R + \sigma\left(x_1 - x_2\right)}{\ell} \tag{8}$$

Where, ρ is resistance gradient of sample wire of length ℓ, σ is resistance per unit length of bridge wire in Ω/cm, R is known resistance in Ω. $x_1$ and $x_2$ are balancing lengths before and after interchanging the known resistances in Carey Foster apparatus.

***2.3.4. Adsorption Isotherm Measurements***

For surface study and to determine the mechanism of adsorption of inhibitor on mild steel surface, the extent of adsorption i,e concentration of inhibitor per unit surface covered (C/θ) was related to the concentration of inhibitor. Different adsorption isotherms were tested from linear regression coefficients, calculated using Fortran 2.0 software. Adsorption constant ($K_{ads}$) and Gibb's free energy change of adsorption ($\Delta G$) were calculated by using equations 9, 10 and 11.

$$C/\theta = K_{ads} C^{1/n} \quad \text{(Freundlich)} \tag{9}$$

$$\theta = \frac{K_{ads} C}{1 + K_{ads} C} \quad \text{(Langmuir)} \tag{10}$$

$$\Delta G = -RT \ln K_{ads} \tag{11}$$

Where *n* is the degree of adsorption.

### *Kinetics and Thermodynamic Measurements*

Rate Constant of chemisorption can be determined from *Eyring equation* given below

$$k = \frac{k_B T K_{ads}}{h} \tag{12}$$

Where, $k$ is rate constant of adsorption at temperature T, $k_B$ is Boltzmann Constant and h is Plank's Constant.

With the help of rate constant of chemisorption of inhibitor on the mild steel surface, activation energy, enthalpy and entropy of adsorption can be calculated from equations 13 and 14.

$$\ln \frac{k_2}{k_1} = \frac{E_a}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \tag{13}$$

and

$$\ln K_{ads} = -\frac{\Delta H_{ads}}{RT} + \frac{\Delta S_{ads}}{R} \tag{14}$$

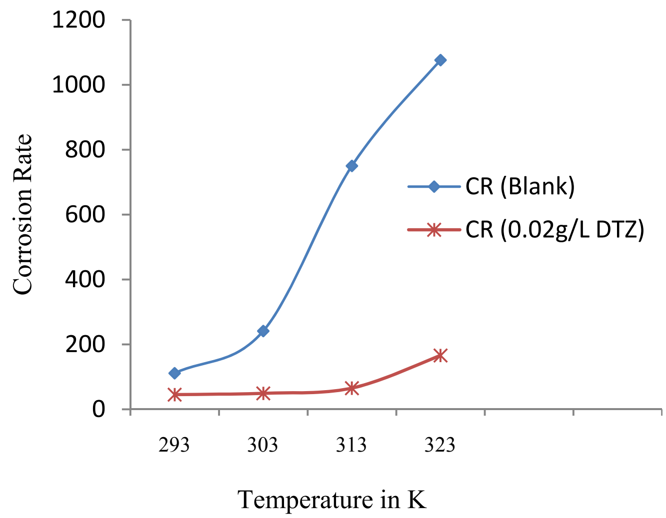
Where, $R$ is gas constant, $\Delta H_{ads}$ and $\Delta S_{ads}$ are enthalpy and entropy of chemisorption of inhibitor on mild steel surface.

## Experimental Work

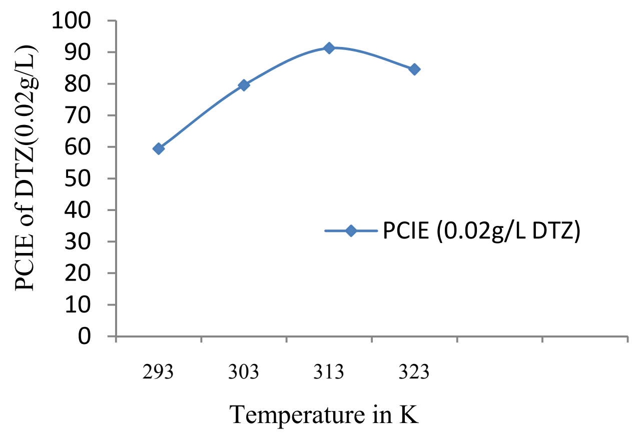
### Gravimetric Study

### *Effect of Temperature*

A non linear increase of corrosion rate is reported during the corrosion testing of mild steel sample wire in acidic solution without DTZ, which, initially increases at a slow rate at low temperature and then, vigorously at high temperatures. Addition of 0.02g/L of DTZ decreased the rate of corrosion tremendously. Effectiveness of DTZ as corrosion inhibitor is witnessed by Figure 1 (a) and (b). A bell shaped graph of PCIE of DTZ gives clue about a polynomial dependence of PCIE on temperature. DTZ gives a peak of PCIE at 313 K, which might be due to diffusion of acid into the adsorbed DTZ film on the surface of mild steel at elevated temperatures and slow adsorption rate at low temperatures.




(a)




(b)

Figure 1 (a): Variation of Corrosion Rate of Mild Steel Wire in absence and presence of DTZ at 0.02g/L Concentration and (b) Variation of Percentage Corrosion Inhibition Efficiency of DTZ at 0.02g/L Concentration with Temperature in 1.0M HCl solution.

***Effect of Concentration***

Sharp decrease in corrosion rate with the increase in concentration of inhibitor from 0.02g/L to 0.04 g/L, and constancy, at all temperatures, in acidic solution, proves (i) the inhibition action of DTZ at all temperatures under consideration (ii) adsorption of DTZ on the surface of mild steel and shift in CR values at, a particular concentration, with increase in temperature, depicts the increase in mobility and hence reactivity of corrosion activators.

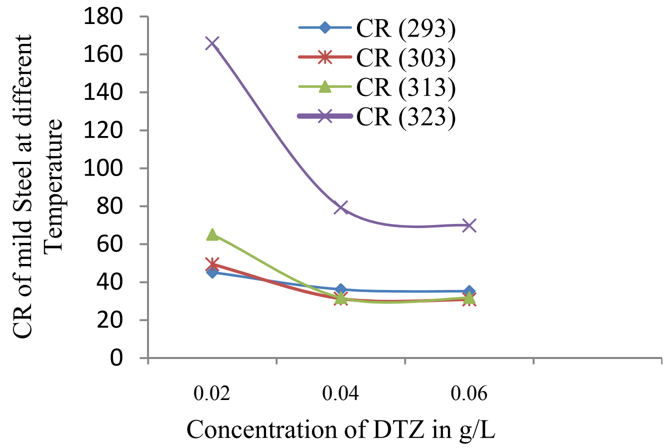



Figure 3: Variation of Corrosion Rate (at different Temperatures) of Mild Steel Wire in absence and presence of different concentration of DTZ in 1.0M HCl solution.

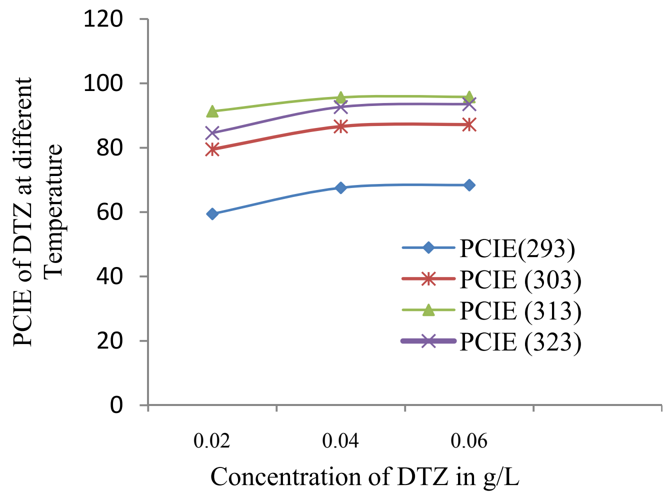



Figure 4: Variation of Percentage Corrosion Inhibition Efficiency of DTZ (at different Temperatures) of Mild Steel Wire in absence and presence of different concentration of DTZ in 1.0M HCl solution.

**Mechanical Study**

Results of different elastic coefficients were recorded to determine the inhibition action of DTZ on mild steel samples. Shear modulus and Young's modulus of each sample were recorded by measuring time period of 15 oscillations one by one on Maxwell's Needle apparatus and Searle's apparatus. From these elastic coefficients bulk modulus and Poisson's ratio were calculated. Results of all elastic constants are summarized in Table 1 and Figure 5-7. Results of mechanical study revealed that all the elastic constants of mild steel sample wires decreased with the corrosion rate and increased with increased concentration of DTZ. A drastic decrease in volume elasticity was noticed at high corrosion rate due to high volumetric strain which is possibly due hydrogen gas embrittlement by action of acid on the mild steel.

Table 2: Results of different Elastic coefficients and parameters in different concentrations of DTZ at different Temperatures

| Temperature | Concentration of DTZ | Young's Modulus (Y) | Modulus of Rigidity (η) | Bulk Modulus (K) | Poisson's Ratio (σ) |
|---|---|---|---|---|---|
| 293K | Blank | 212.5 | 78.73 | 235.5 | 0.3496 |
| | 0.02g/L | 226.7 | 82.18 | 314.7 | 0.3792 |
| | 0.04g/L | 227.5 | 83.43 | 315.7 | 0.3799 |
| | 0.06g/L | 227.9 | 82.56 | 317.1 | 0.3802 |
| 303K | Blank | 210.7 | 78.20 | 229.8 | 0.3472 |
| | 0.02g/L | 226.4 | 82.12 | 310.3 | 0.3784 |
| | 0.04g/L | 228.1 | 82.51 | 322.7 | 0.3822 |
| | 0.06g/L | 229.9 | 83.11 | 327.8 | 0.3831 |
| 313K | Blank | 207.8 | 77.07 | 226.9 | 0.3480 |
| | 0.02g/L | 224.6 | 81.78 | 294.9 | 0.3731 |
| | 0.04g/L | 228.4 | 82.63 | 322.6 | 0.3820 |
| | 0.06g/L | 229.5 | 82.97 | 326.6 | 0.3829 |
| 323K | Blank | 201.2 | 74.7 | 218.7 | 0.3467 |
| | 0.02g/L | 214.0 | 79.2 | 239.4 | 0.3510 |
| | 0.04g/L | 219.0 | 80.9 | 249.8 | 0.3541 |
| | 0.06g/L | 222.2 | 81.1 | 289.9 | 0.3721 |

Interaction of acid increased the porosity, weakened the metallic bond, decreased the strength and swallowed the mild steel specimen wires from which sample wires became soft. Poisson's ratio increased due to decrease in longitudinal strain and increased in lateral strain due to inhibition action of dithizone and reduction of loss of mechanical strength of mild steel.

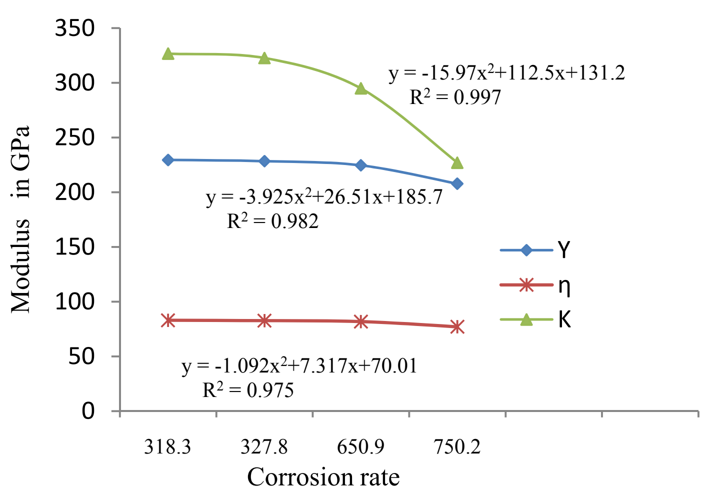



Figure 5: Variation of Young's Modulus, Shear Modulus and Bulk Modulus of sample wires with Corrosion Rate in presence of DTZ in 1.0 M HCl solution at 40°C.

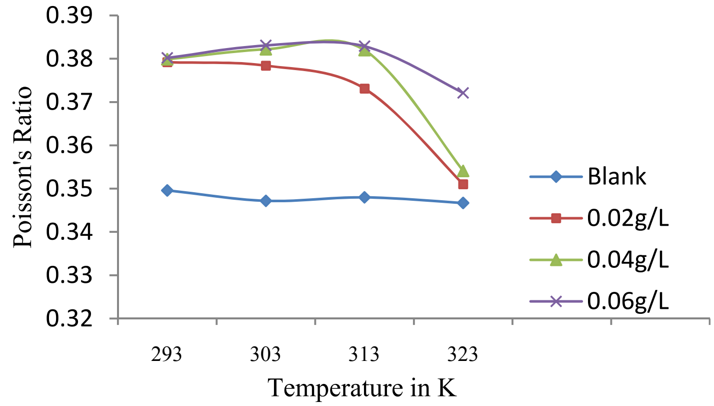



Figure 6: Variation of Poisson's Ratio of sample wires with Temperature in presence of different concentrations of DTZ in 1.0 M HCl solution.

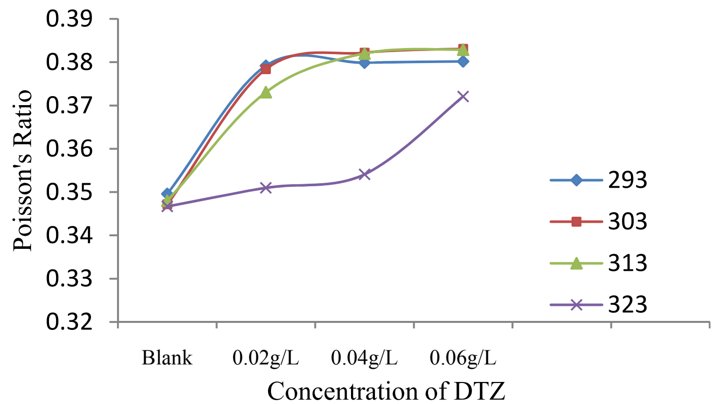



Figure 7: Variation of Poisson's Ratio of sample wires with concentration of DTZ in 1.0 M HCl solution at different Temperatures.

## Electrical Study-

Results obtained by the study of electrical resistance gradient are shown in Table 2 and Figure 8. It is observed that opposition offered by the sample wire to the flow of current through itself is decreasing with the increase in concentration of inhibitor in HCl solution. This might be due to the decrease in surface erosion of the wire occurring due to corrosion. High resistance of blank sample suggests that the corrosion products formed on the surface of wire are of poor electric conductivity and have decreased the effective cross section area and skin effect, which caused the high resistance of blank sample. Protective film formed by inhibitor on sample wire barred the entry of acid ions into the crystal structure and decelerated the rate of corrosion.

Table 2: Resistance Gradient (Ω/cm) of sample wire in different concentrations of DTZ

| Concentration | 293K | 303K | 313K | 323K |
|---|---|---|---|---|
| Blank | 0.146 | 0.172 | 0.203 | 0.225 |
| 0.02g/L | 0.117 | 0.119 | 0.125 | 0.159 |
| 0.04g/L | 0.105 | 0.099 | 0.099 | 0.134 |
| 0.06g/L | 0.101 | 0.097 | 0.098 | 0.129 |

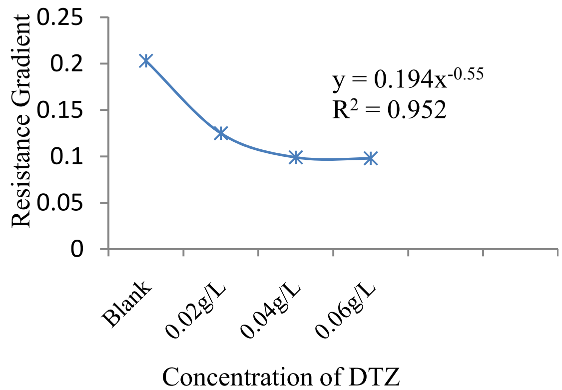



Figure 8: Variation of Poisson's Ratio of sample wires with different concentration of DTZ in 1.0 M HCl solution at 40°C.

**Surface Study-**

From the result obtained from adsorption isotherm analysis, Freundlich and Langmuir adsorption isotherms were tested as given in Figure 9 and Figure 10 on the basis of regression coefficients calculated by Fortran 2.0 which were found to be best fit i,e found very close to unity. Adsorption constant ($K_{ads}$) and Gibb's free energy change of adsorption ($\Delta G$) from both adsorption isotherms were calculated which are given in Table 3.

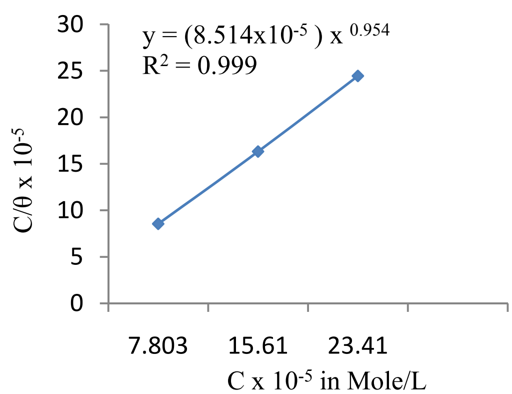



Figure 9: Freundlich Adsorption Isotherm of DTZ onto mild steel surface in 1.0M HCl solution at 40°C.

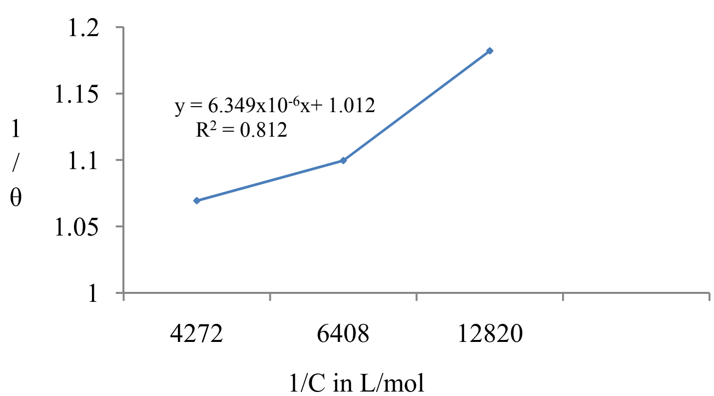



Figure 10: Langmuir Adsorption Isotherm of DTZ onto mild steel surface in 1.0M HCl solution at 40°C.

Table 3: Freundlich and Langmuir adsorption isotherm parameters obtained from corrosion test in 1.0M HCl solution containing 0.02g/L DTZ at 40$^o$C.

| Parameters | Freundich Adsorption Isotherm | Langmuir Adsorption Isotherm |
|---|---|---|
| Slope | - | 6.349 x $10^{-6}$ |
| n | 1.101 | - |
| $K_{ads}$ | 8.514 x $10^{-5}$ | 15.751 x $10^4$ |
| ΔG | +24.39 KJ/mol | -31.14 KJ/mol |

Adsorption constants and Gibb's free energy change data obtained from both adsorption isotherms clearly indicated that the Langmuir adsorption isotherm was found to be best fit in adsorption of DTZ as corrosion inhibitor on mild steel sample wires. It is assumed that the mild steel surface contains a fixed number of adsorption sites and each site chemically holds one inhibitor molecule by interaction of lone pairs of N and S atoms and π electron delocalized systems of DTZ with the d-orbitals of metals present in mild steel. Very high negative Gibb's free energy clearly indicates the spontaneous chemisorption of DTZ on mild steel surface. Almost independence of observed extent of adsorption at very high concentration is due to non-availability of adsorption sites on the mild steel surface for adsorption of inhibitor.

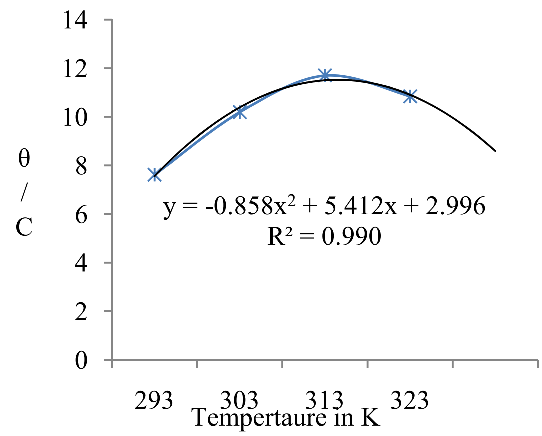



Figure 11: Adsorption Isobar of 0.02g/L DTZ onto mild steel surface in 1.0M HCl solution.

**Kinetics and Thermodynamic Study**

Rise in temperature provides the activation energy of adsorption and the rate of adsorption elevates upto a limit. Bell shaped adsorption isobar and negative Gibb's free energy change for adsorption confirms the chemisorption of DTZ on mild steel surface (Figure 11 and Table 4).

Table 4: Kinetic study data obtained from corrosion test in 1.0M HCl solution containing 0.02g/L DTZ at different temperature.

| Temperature (K) | Adrsorption Constant ($K_{ads}$ x$10^4$) | Rate Constant ($k_{ads}$ x$10^{17}$) | Activation Energy ($E_a$ in KJ/mol) | Gibb's Free Energy Change ($\Delta G_{ads}$ in KJ/mol) |
|---|---|---|---|---|
| 293 | 0.43 | 2.251 | - | -20.37 |
| 303 | 7.35 | 4.492 | + 51.04 | -28.23 |
| 313 | 15.74 | 9.620 | + 55.37 | -31.14 |
| 323 | 7.58 | 4.630 | +18.91 | -30.17 |

Table 5: Thermodynamic study data obtained from corrosion test in 1.0M HCl solution containing 0.02g/L DTZ at different temperature.

| Slope | Intercept | Enthalpy Change (ΔH) | Entropy Change (ΔS) |
|---|---|---|---|
| $-2.887 \times 10^3$ | 20.615 | 24.027 KJ/mol | 171.35 J/mol |

Kinetics and thermodynamic study reveals that the rate constant of adsorption increases and adsorption becomes more spontaneous upto a temperature limit by increase in temperature. At high temperature, adsorption-desorption equilibrium shifts towards backward endothermic desorption process and desorption becomes dominant over adsorption (Table 5). Because of sufficient energy at high temperature, chemical interaction of chloride ions increases with adsorbed film and chloride ions penetrate into the film on mild steel surface by which desorption process becomes fasy and corrosion rate increases.

## Conclusions

Following conclusions are drawn from the study of corrosion inhibition of mild steel sample wires in different concentrations of DTZ, at different temperatures:

i. DTZ shows very strong corrosion inhibition action due to interaction of lone pairs of N and S atoms and π electron systems of DTZ with the d-orbitals of metals present in mild steel according to Lewis acid-base interaction.
ii. CR is significantly decreased and PCIE is tremendously increased with increase in the concentration of DTZ in HCl solution initially but this effect is poorly observed at very high concentration of inhibitor and very high temperature due to non-availability of adsorption active sites on the mild steel surface and increased activity of corrosion activators respectively.
iii. Young modulus, Shear modulus and Bulk modulus of mild steel sample wires increased with increased concentration of DTZ due to increase in the porosity, weakening of metallic bond and swallowing of mild steel by action of acid and decreased non significantly at elevated temperatures.
iv. Poisson's ratio increased due to decrease in longitudinal strain and increased in lateral strain due to inhibition action of DTZ and reduction of loss of mechanical strength of mild steel.
v. High resistance of blank sample suggests that the corrosion products formed on the surface of wire are of poor electric conductivity and have decreased the effective cross section area and skin effect, which caused the high resistance of blank sample.
vi. Langmuir adsorption isotherm was found to be best fit in adsorption of DTZ on mild steel. Very high negative Gibb's free energy indicates the spontaneous chemisorption of DTZ on mild steel surface.
vii. Kinetics and thermodynamic studies reveals that rate constant of adsorption is increased and adsorption becomes more spontaneous upto a temperature limit by increase in temperature. At high temperature, adsorption-desorption equilibrium shifts toward backward endothermic desorption process and desorption become dominant over adsorption.
viii. At high temperature chemical interaction of chloride ions become high with adsorbed film and chloride ions penetrate the film on mild steel surface by which desorption process and corrosion rate become high.

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