

STUDY OF INHIBITION EFFECT OF THE CORROSION OF MILD STEEL IN 1 N HCl ACID SOLUTION BY VANILLINTHIOSEMICARBAZONE, VANILLIN-4-METHYLTHIOSEMICARBAZONE AND VANILLIN-4-ETHYLTHIOSEMICARBAZONE

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ABSTRACT

Vanillinthiosemicarbazone and its methyl, ethyl derivatives were tested for their corrosion inhibition towards mild steel in 1 N HCl solution at 298 K using weight loss, Tafel polarization and electrochemical impedance spectroscopy techniques. Potentiodynamic polarization and EIS techniques were applied to study the metal corrosion behavior in the absence and presence of different concentrations of these inhibitors under the influence of various experimental conditions. These inhibitors revealed good corrosion inhibition efficiency even at low concentrations like 1×10^{-3} M towards mild steel in HCl medium. Comparison of corrosion inhibition efficiencies of the derivatives and their parent were also investigated. Polarization curves showed that the three studied compounds act as mixed type inhibitors. The effects of chemical structure of the three tested inhibitors were discussed. Results obtained from OCP versus time, polarization and impedance measurements are in good agreement. Studies showed all the three compounds Vanillinthiosemicarbazone (VTSC), Vanillin-4-methylthiosemicarbazone (VMTSC) and Vanillin-4-ethylthiosemi-carbazone (VETSC) on mild steel surface to act as mixed type inhibitors and to obey the Langmuir adsorption isotherm.

Keywords: Mild steel, Vanillinthiosemicarbazone (VTSC), Vanillin-4-methylthiosemicarbazone (VMTSC), Vanillin-4-ethylthio semicarbazone (VETSC) Potentiodynamic polarization, Electrochemical impedance spectroscopy (EIS) and Scanning electron microscopy (SEM)

I. INTRODUCTION

Acid solutions are generally used for the removal of rust and scale in industrial processes. Hydrochloric acid is widely used in the pickling, cleaning and descaling of steel and ferrous alloys. Severe corrosion problems arise due to the wide use of hydrochloric acid for pickling, rescaling and cleaning processes of mild steel (MS) surfaces. Inhibitors are used to control the corrosion of metallic materials by controlling metal dissolution and consumption. Most acid inhibitors are organic compounds containing nitrogen, sulphur or oxygen atoms or N-heterocyclic compounds with polar groups and/or pi-electrons [1, 2, 8 & 10]. The primary step in the action of organic corrosion inhibitors in acid solutions is usually adsorption at the metal-solution interface. The adsorption process depends on the electronic characteristic of the molecules (adsorbate), the chemical composition of the solution, and nature of the metal surface, temperature of the reaction and on the electrochemical potential at the metal-solution interface. Many studies have been made on the corrosion and inhibition of steels in acid media. A few research reports revealed that the inhibition efficiency

of Schiff's bases is much higher than that of corresponding aldehydes and amines, and this may be due to the presence of a $>C = N -$ group in the molecules [8]. The choices of these compounds were based on molecular structure with delocalized pi electrons (aromaticity), a thiosemicarbazone moiety [1]. Organic inhibitors generally protect the metal from corrosion by forming a film on the metal surface [9].

The adsorption requires the existence of attractive forces between the adsorbate and the metal. According to the type of forces, adsorption can be physisorption, chemisorptions or a combination of both. Physisorption is due to electrostatic attractive forces between inhibiting organic ions or dipoles and the electrically charged surface of the metal. Chemisorption is due to the interaction between unshared electron pairs or electrons of the adsorbate with the metal in order to form a coordinate type of bond. It may take place in presence of heteroatom (P, S_e, S, N, O), with lone-pair electrons and/or aromatic rings in the adsorbed molecules [11, 12 & 14]. The main objective of the present research paper was to study the inhibition action of newly synthesized Schiff's bases VTSC,

VMTSC and VETSC on the corrosion of mild steel in 1.0 N HCl at 298 K and to compare their protection powers. These structural features favor the interaction of the inhibitors VTSC, VMTSC and VETSC with mild steel.

II. EXPERIMENTAL DETAILS

A. Materials

Mild steel samples of the composition, C = 0.058%, Mn = 0.252%, P = 0.004%, S = 0.002% and Fe = 99.684% were used for the measurement of the corrosion rates. Rectangular specimens of working surface area $5 \times 2.5 \times 0.16 \text{ cm}^2$ were used for the weight loss method. These specimens were polished mechanically using emery papers of different grades and washed thoroughly with triple distilled water, degreased with acetone and dried using air flow at room temperature.

The aggressive solution (1.0 M HCl) was prepared by dilution of analytical grade 37% HCl solution with double-distilled water [1].

B. Inhibitors

Vanillinthiosemicarbazone (VTSC) Vanillin-4-methylthiosemicarbazone (VMTSC) and Vanillin-4-ethylthiosemicarbazone (VETSC) were prepared by the condensation of Thiosemicarbazide, 4-methylthio semicarbazide and 4-ethylthiosemicarbazide with vanillin (Sigma-Aldrich). The structures of the Schiff bases are shown in Fig.1.a, 1.b and 1.c. The inhibitors VTSC, VMTSC and VETSC were used in concentrations such as $1 \times 10^{-4} \text{ M}$, $2.5 \times 10^{-4} \text{ M}$, $5 \times 10^{-4} \text{ M}$, $7.5 \times 10^{-4} \text{ M}$ and $10 \times 10^{-4} \text{ M}$.

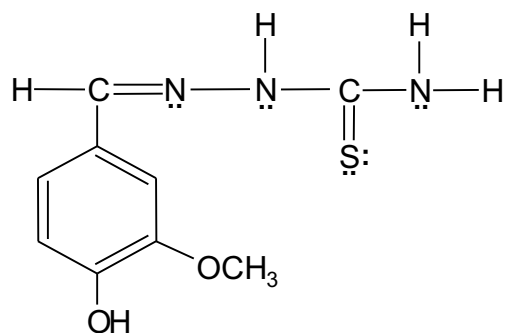


Fig. 1. (a) Structure of Vanillinthiosemicarbazone (VTSC)

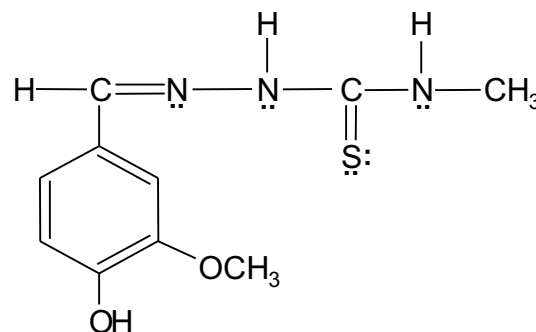


Fig. 1. (b) Structure of Vanillin-4-methylthiosemicarbazone (VMTSC)

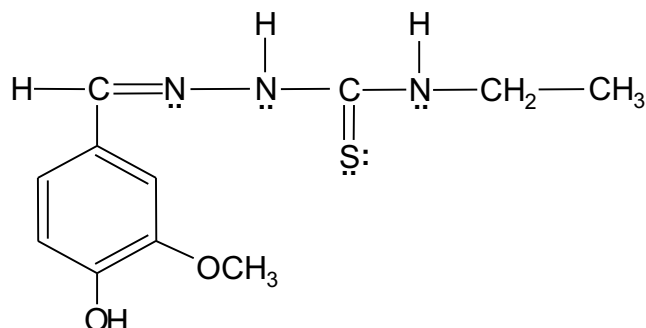


Fig. 1. (c) Structure of Vanillin-4-ethylthiosemicarbazone (VETSC)

C. Weight loss method

Although there are many experimental techniques which can be used to evaluate the inhibitor efficiencies, weight loss is one of the simplest and frequently used methods [9].

100 ml of 1N HCl without inhibitor was used as blank test solution. These MS coupons were immersed in hanging position in different concentrations of inhibitor solutions taken in 100 ml beaker and weight losses of the metal coupons were noted. After the corrosion test, the specimens of steel were carefully washed in double-distilled water, dried and then weighed. The rinse removed loose segments of the film of the corroded samples. Triplicate experiments were performed in each case and the mean value of the weight loss is reported. Weight loss allowed us to calculate the mean corrosion rate as expressed in $\text{mg cm}^2 \text{ h}^{-1}$ [16, 17].

D. Electrochemical methods

Tafel polarization curves were obtained with a scan rate of 5 mV/S in the potential range from $\pm 250 \text{ mV}$ relative to the open circuit potential. Polarization

measurements were performed using a computer-controlled CH instrument 660B electrochemical work station. A cell containing three compartments of electrodes was used. The working mild steel electrode was embedded in Araldite to offer its cross-sectional area (1 cm^2) in contact with the solution. A saturated calomel electrode (SCE) and a platinum electrode were used as the reference and the counter electrodes, respectively. All the potentials were reported versus that of the SCE. Corrosion current density values were obtained by Tafel extrapolation method.

EIS measurements were performed at open circuit potential in the frequency range from 1 Hz to 100000 Hz with a 5 mV sine wave as excitation signal. The same cell and instrument as in the polarization method were used. The double layer capacitance (C_d) and resistance polarization (R_p) were calculated from Nyquist plots as described elsewhere [2].

E. SEM analysis

Scanning electron microscopy (SEM) was used to study the surface morphology of the mild steel.

(InstrumentName = S – 3400,

SerialNumber = 340745 – 05SEM).

Polished mild steel specimens were tested in plain 1N HCl solution and maximum inhibitor concentration 10.0×10^{-4} M in 1N HCl solution. Then

they were washed with distilled water, dried, kept in desiccators and were subjected to scanning electron microscopic examination.

III. RESULT AND DISCUSSION

A. Weight loss method

The inhibition efficiency (%) was calculated by the following relation:

$$I.E = \left(\frac{W_o - W}{W_o} \right) \times 100\% \quad (1)$$

Where W_o and W are weight losses in the absence and presence of inhibitor, respectively [1]. The weight loss of mild steel specimen in 1 N HCl solution and solutions containing different concentrations of VTSC, VMTSC and VETSC were determined during 2-h immersion [2]. The coupons immersed in VTSC inhibitor solutions showed minimum weight loss. VTSC reduced the corrosion rate to a significant extent and thereby showing higher inhibition efficiencies. Table.1 gives information about corrosion rate, inhibition efficiency with concentration of the synthesized inhibitors. For inhibitor concentrations starting from 1×10^{-4} M on-wards, the achieved inhibition efficiencies are above the prescribed efficiency limit of 87% for acid pickling inhibitors [2]. It can be seen that the inhibition efficiency increases with increase in concentration of inhibitors [16], which suggests that inhibition is a result of adsorption of inhibitor on the metal surface.

Table 1. Weight loss data of mild steel in 1N HCl for various concentrations of VTSC, VETSC and VMTSC

Inhibitor concentration (M)	Corrosion rate ($\text{mg}/\text{cm}^2/\text{min}$)			Inhibition efficiency (%)		
	VTSC	VETSC	VMTSC	VTSC	VETSC	VMTSC
Blank (in HCl)	0.026	0.026	0.026	–	–	–
1×10^{-4}	0.019	0.0158	0.0114	26.34	39.39	56.17
2.5×10^{-4}	0.0096	0.0081	0.0092	63.07	68.84	64.61
5×10^{-4}	0.0086	0.0078	0.0087	66.89	70	66.4
7.5×10^{-4}	0.0043	0.0054	0.0075	83.21	79.02	71.09
1×10^{-3}	0.0019	0.0024	0.0038	92.5	90.67	85.08

B. Polarisation Technique

Figures 1, 2 and 3 shows the cathodic and anodic polarization curves recorded for mild steel in aerated stagnant 1.0 N HCl solutions without and with various concentrations of VTSC, VMTSC and VETSC at a scan rate of 1.0 m VS^{-1} at 30°C . The values of the corrosion current density (i_{corr}) for mild steel corrosion reaction without and with inhibitors were determined, therefore by extrapolation of the cathodic and anodic Tafel lines to the corrosion potential (E_{corr}). The cathodic as well as the anodic reactions are markedly affected by the inhibitor toward lower current densities. Based on that there is no definite shift in the corrosion potential and the marked decrease of the current density upon introducing the inhibitors in the aggressive solution. All the inhibitors are considered as mixed-type inhibitor. These observations demonstrate that the addition of the inhibitors reduces mainly the cathodic process and also retards the anodic process. The corrosion current density (i_{corr}) decreased when the concentration of the inhibitors were increased [6, 14].

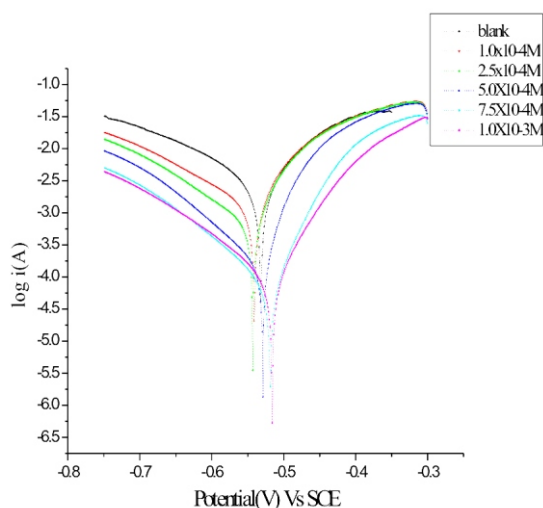


Fig 1. Typical Tafel plots for mild steel in 1N HCl containing different concentrations of VTSC

This indicates the inhibiting effect of VTSC, VMTSC and VETSC on the acid corrosion of mild steel. Electrochemical kinetic parameters, protection efficiencies (IE_p (%)) and rates of corrosion associated with potentiodynamic polarization measurements recorded for mild steel in 1.0 N solutions in absence

and presence of various concentrations of VTSC, VMTSC and VETSC are given in Table 2.

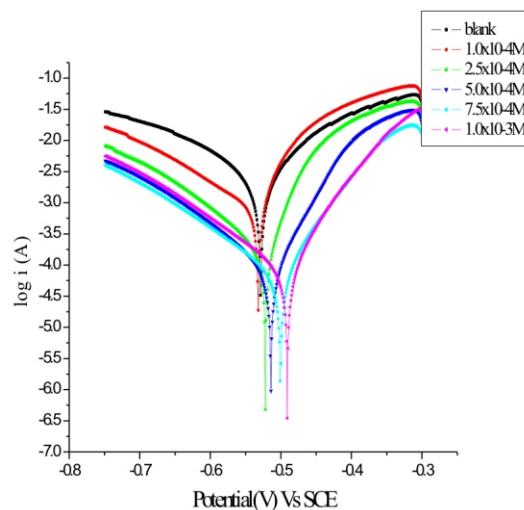


Fig 2. Typical Tafel plots for mild steel in 1N HCl containing different concentrations of VETSC

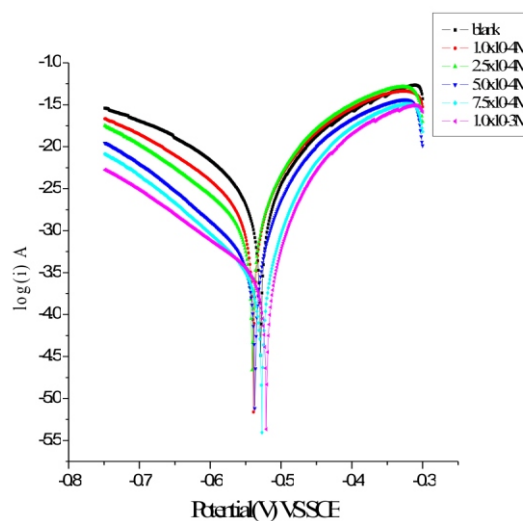


Fig 3. Typical Tafel plots for mild steel in 1N HCl containing different concentrations of VMTSC

$IE\%$ were calculated from polarization measurements according to the equation given

$$IE(\%) = \left(\frac{i_{corr} - i_{corr}(inh)}{i_{corr}} \right) \times 100 \quad (3)$$

C. Electrochemical Impedance Studies

Results obtained from EIS measurements for mild steel in 1.0 N HCl solution in the absence and presence of different concentrations of VTSC, VMTSC

Table 2: Potentiodynamic polarization parameters of mild steel in 1 M HCl for various concentrations of VTSC, VETSC and VMTSC

Inhibitor concentration (M)	$R_{et} (\Omega \text{ cm}^2)$			$C_{dl} (\mu F)$			$I.E (%)$		
	VTSC	VETSC	VMTSC	VTSC	VETSC	VMTSC	VTSC	VETSC	VMTSC
Blank (1 M HCl)	23	23	15	1.045	1.045	1.045	–	–	–
1.0×10^{-4}	100	105	55	11.92	4.867	2.97	77	78.09	72.72
2.5×10^{-4}	330	350	98	21.03	9.27	3.56	93	93.42	84.69
5.0×10^{-4}	370	450	118	70.39	87.6	6.08	93.8	94.88	87.28
7.5×10^{-4}	470	537	149	105.6	13.31	10.49	95.1	95.71	89.93
1.0×10^{-3}	550	562	159	42.99	1290	60.18	96.8	95.9	90.56

Table 3. Electrochemical impedance measurement of mild steel in 1M HCl for various concentrations of VTSC, VETSC and VMTSC

Inhibitor concentration (M)	$R_{et} (\Omega \text{ cm}^2)$			$C_{dl} (\mu F)$			$I.E (%)$		
	VTSC	TETSC	VMTSC	VTSC	VETSC	VMTSC	VTSC	VETSC	VMTSC
Blank (1M HCl)	23	23	15	1.045	1.045	1.045	–	–	–
1.0×10^{-4}	100	105	55	11.92	4.867	2.97	77	78.09	72.72
2.5×10^{-4}	330	350	98	21.03	9.27	3.56	93.03	93.42	64.69
5.0×10^{-4}	370	450	118	70.39	87.6	6.08	93.78	94.88	87.28
7.5×10^{-4}	470	537	149	106.6	13.31	10.49	95.1	95.71	89.93
1.0×10^{-3}	550	562	159	42.99	12.90	60.18	95.81	95.9	90.56

and VETSC were presented in the form of Nyquist (Fig. 4, 5 and 6) plots. The plots showed a depressed capacitive loop which arises from the time constant of the electrical double layer and charge transfer resistance. The impedance of the inhibited mild steel increases with increase in the inhibitor concentration and consequently the inhibition efficiency increased. Inhibitor molecules get adsorbed on the mild steel/acid solution interface and thereby produce a barrier for the metal to diffuse out to the bulk and this barrier increases with increasing the inhibitor concentration. The values of the polarization resistance were

calculated by subtracting the high frequency intersection from the low frequency intersection. Double layer capacitance values were obtained at maximum frequency (f_{max}), at which the imaginary component of the Nyquist plot is maximum, and calculated using the following equation.

$$C_{dl} = \frac{1}{2\pi f_{max}} \times \frac{1}{R_p} \quad (2)$$

Increase of the polarization resistance and decrease of the double layer capacity with increasing inhibitor concentration indicate that these compounds

inhibit the corrosion rate of mild steel by an adsorption mechanism. The various data's obtained are tabulated in Table 3.

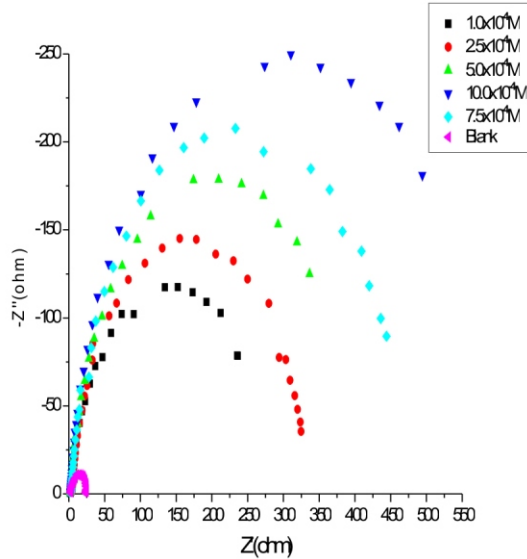


Fig. 4 Typical Nyquist plots obtained for MS in 1M HCl in the presence and absence of different concentrations of VTSC at 300 K

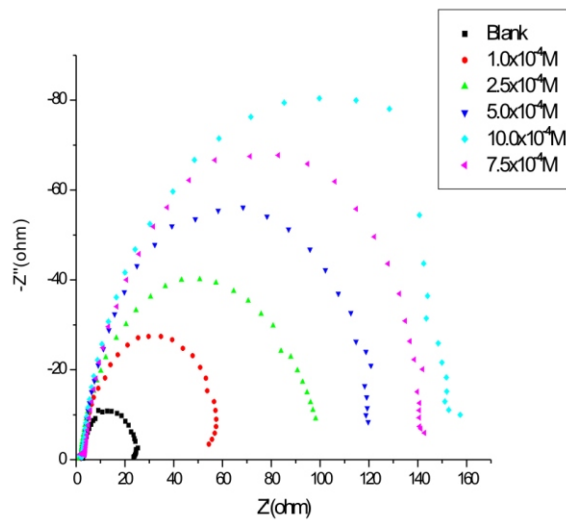


Fig. 5 Typical Nyquist plots obtained for MS in 1M HCl in the presence and absence of different concentrations of VMTSC at 300 K

In fact, the presence of all three inhibitor compounds enhances the value of the polarization resistance in the acidic solution the effect being more pronounced with compound III. Values of double layer capacitance are also brought down to the maximum

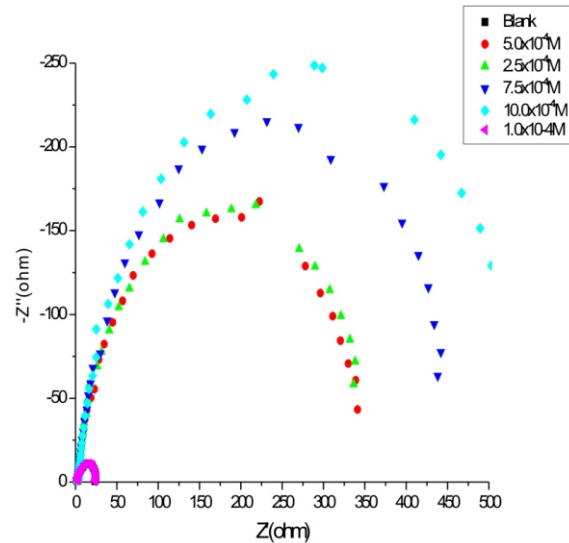


Fig. 6 Typical Nyquist plots obtained for MS in 1M HCl in the presence and absence of different concentrations of VTSC at 300 K

extent in the presence of all three compounds, the decrease being more effective in compound III, and the decrease in the values of C_{dl} follow the order similar to that obtained for the I_{corr} studies. The decrease in is due to the adsorption of this compound on the metal surface leading to the formation of a surface film in the acidic solution [12].

An alternative formula of the Arrhenius equation is the transition state equation:

$$C_R = \frac{RT}{Nh} \exp\left(-\frac{\Delta H^*}{RT}\right) \exp\left(\frac{\Delta S^*}{R}\right) \quad (4)$$

From impedance measurement results, the percentage inhibition efficiency ($I.E$) was calculated using the expression

$$I.E = \frac{R_{ct} - R'_{ct}}{R_{ct}} \times 100\% \quad (5)$$

where R_{ct} and R'_{ct} denote charge transfer resistance of electrode with and without inhibitor, respectively [6].

D. SEM study

The SEM micrographs were recorded (Fig. 7) to establish the interaction of inhibitor molecules with metal surface. The SEM micrographs showed features of the mild steel surface after 2 hr in 1.0 N HCl in the absence and presence of 0.001 M VTSC, VMTSC and

VETSC at 298 K. The SEM micrographs revealed that the mild steel specimen immersed in inhibited solution was in better condition having smooth surface while the metal surface immersed in one molar hydrochloric acid solution was rough covered with corrosion products and appeared like full of pits and cavities. These indicated that the above inhibitors hindered dissolution of the mild steel by forming protective film on the metal surface and thereby reduce the corrosion rate.

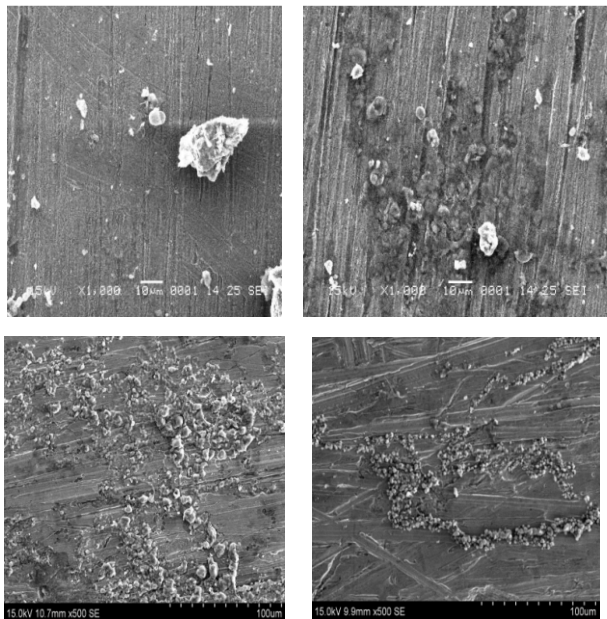


Fig. 7. SEM Microscopic Images

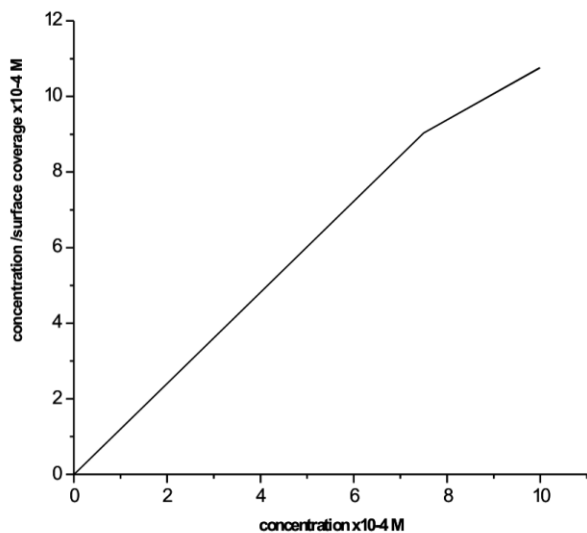


Fig. 8. Langmuir adsorption plot for mild steel in 1 M HCl containing different concentration of VTSC at 300 K

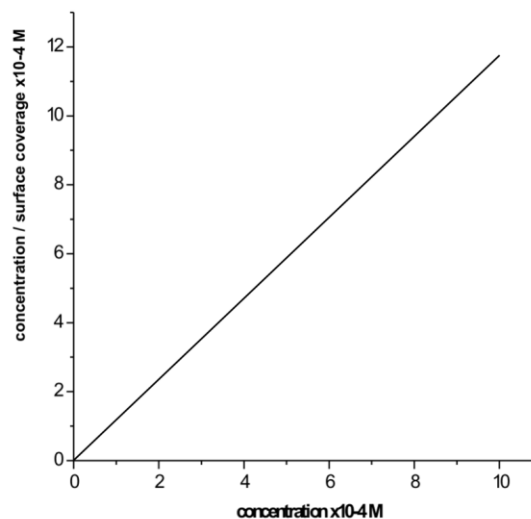


Fig. 9. Langmuir adsorption plot for mild steel in 1 M HCl containing different concentration of VETSC at 300 K

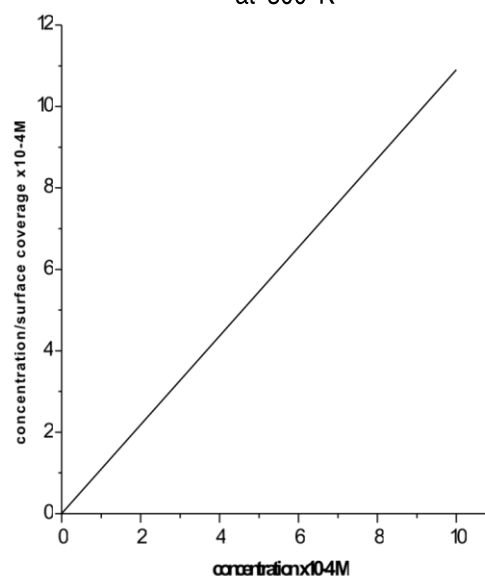


Fig. 10. Langmuir adsorption plot for mild steel in 1M HCl containing different concentration of VMTSC at 300 K

E. Adsorption Isotherm

The interactions between the inhibitor and the mild steel surface can be examined by the adsorption isotherm [6, 7]. The standard free energy of adsorption has been calculated from the equation

$$K_{eq} = \frac{1}{55.5} \exp \left(\frac{\Delta G_{ads}}{RT} \right) \quad (6)$$

Where K_{eq} is equilibrium constant [7, 8].

where C is the molar concentration of inhibitor, K_{ads} is the equilibrium constant of the adsorption process and θ is the degree of coverage by inhibitor molecules on the metal surface, calculated from the relationship

$$\theta = \frac{W_o - W}{W_o} \quad (7)$$

Where W_o and W are mass losses of mild steel in uninhibited and inhibited solutions. Adsorption equilibrium constant (K_{ads}) and free energy of adsorption (ΔG_{ads}) were calculated using the relationships

$$K_{ads} = \frac{1}{C} \times \frac{\theta}{1 - \theta} \quad (8)$$

$$\Delta G_{ads} = -2.303 RT \log (55.5 K_{ads}) \quad (9)$$

At, 300 K, the free energy of adsorption (ΔG_{ads}) and adsorption equilibrium constant (K_{ads}) are found to be, -13.56 , 13.29 and -13.55 KJ/mole & 5.35×10^3 , 5.6×10^3 and 5.3×10^3 for VTSC, VMTSC and VETSC respectively. The negative and low value of ΔG_{ads} indicates the spontaneous and physical nature of adsorption.

The inhibition efficiency afforded by the inhibitor molecules to mild steel due to the presence of electron rich N and aromatic rings. The adsorption of the inhibitor molecules on the mild steel surface can be explained on the basis of the donor acceptor inter-action between p electrons of donor atoms N and aromatic rings of the inhibitors and the vacant d orbital's of iron surface atoms.

It is assumed that organic inhibitors establish inhibition by way of adsorption onto the metal surface. The adsorption processes of inhibitors are influenced by the chemical structures of organic compounds, the nature and surface charge of metal, the distribution of charge in molecule [9].

IV. CONCLUSION

1. The inhibition efficiencies of VTSC, VMTSC and VETSC increases with an increase in inhibitor concentration.

2. The inhibitors VTSC, VMTSC & VETSC showed maximum inhibition efficiency of 97.12, 98 and 99.75% at concentration.
3. Potentiodynamic polarization proved that VTSC, VMTSC and VETSC are an efficient inhibitor having a mixed type of inhibitor property.
4. Langmuir adsorption isotherm and impedance studies showed that VTSC, VMTSC and VETSC inhibits through its adsorption mechanism.

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