# Thermodynamics and Electrochemical Investigation of (4-(2amino-6-(cyclopropylamino)-9H-purin-9-yl) cyclopent-2-enyl) Methanol Sulphate as Green and Effective Corrosion Inhibitor for Mild Steel in 1M Hydrochloric Acid

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The inhibition effect of new drug namely Abacavir sulfate ((4-(2-amino-6-(cyclopropylamino)-9Hpurin-9-yl) cyclopent-2-enyl) methanol sulphate) on mild steel has been investigated using potentiodynamic polarization, linear polarization (*LPR*), electrochemical impedance spectroscopy (*EIS*) and weight loss measurements. The adsorption of the Abacavir was well described by the Langmuir adsorption isotherm and the adsorption isotherm parameters ( $K_{ads}$ ,  $\Delta G_{ads}$ ) were also determined. Effect of temperature on the efficiency of the corrosion inhibition process was studied and the values of activation energy, pre-exponential factor ( $\lambda$ ), enthalpy of activation and entropy of activation were also calculated to elaborate the mechanism of corrosion inhibition. Tafel polarization measurements revealed that Abacavir is a mixed-type inhibitor. *EIS* plots indicated that the addition of Abacavir increases the charge-transfer resistance ( $R_{ct}$ ), decreases the double-layer capacitance ( $C_{dl}$ ) of the corrosion process. Abacavir showed maximum efficiency 97.7 % at 400 ppm. The *LD*<sub>50</sub> dose for rat is 3200 mg/kg this shows that the drug is non-toxic and environmentally safe.

Keywords: Kinetic parameters; Mild steel; EIS; Acid corrosion, LPR

# **1. INTRODUCTION**

The use of inhibitor is one of the best known methods for protection of mild steel corrosion. The efficiency of inhibitor depends on the nature of environment, nature of metal surface, electrochemical potential at the interface, and the structural feature of inhibitor, which includes number of adsorption centers in the molecule, their charge density, the molecular size, and mode of adsorption, formation of metallic complexes and the projected area of inhibitor on the metal surface [1]. Most well-known acid inhibitors are organic compounds which have  $\pi$  bonds and contains hetero atoms such as sulphur, nitrogen, oxygen and phosphorous which allows the adsorption of compounds on the metal surface [2-6]. The most of the organic inhibitors are toxic, highly expensive and environment unfriendly. Research activities in recent times are geared towards developing the cheap, non-toxic and environment friendly corrosion inhibitors. Thus, the development of the novel corrosion inhibitors of natural source and non-toxic type has been considered to be more important and desirable [7]. Because of their natural origin [8-10], as well as their non-toxic characteristics [11] and negligible negative impacts on the aquatic environment [12], drugs (chemical medicines) seem to be ideal candidates to replace traditional toxic corrosion inhibitors. The literature survey reveals that following drugs were used as efficient corrosion inhibitors for mild steel corrosion in acidic solutions: Ceftriaxone, Cefalexin, Doxycycline, Pheniramine, Fexofenadine, Ceftobiprole, Ranitidine, Rhodanine azosulpha, Dapsone, Mebendazole, Penicillin G, Penicillin V potassium, Ciprofloxacin, Cefadroxil, Ketoconazole, Methocarbamol, Ampicillin, Sparfloxacin, Chloramphenicol, Orphenadrine, Cefotaxime, Cefazolin, Cloxacillin and Rabeprazole Sulfide [13-35].

In the present paper, we describe a systematic study of corrosion of mild steel of Abacavir drug in 1 M HCl using weight loss and electrochemical techniques (*EIS*, potentiodynamic polarization and *LPR*) studies. The IUPAC name of Abacavir is (4-(2-amino-6-(cyclopropylamino)-9H-purin-9-yl) cyclopent-2-enyl) methanol sulphate which have molecular formula,  $C_{14}H_{18}N_6O$  and molecular weight 286.33. Molecular structure and IUPAC name of Abacavir is given in Figure 1. *LD*<sub>50</sub> value for Abacavir is 3200mg/kg for oral dose of rat.



Figure 1. Structure of Abacavir (4-(2-amino-6-(cyclopropylamino)-9H-purin-9-yl) cyclopent-2- enyl) methanol sulfate.

# 2. EXPERIMENTAL

## 2.1 Materials

Mild steel samples having composition (wt %) C = 0.076, Mn = 0.192, P = 0.012, Si = 0.026, Cr = 0.050, Al = 0.023, with Fe- constituting the remaining percentage, were used for gravimetric and

electrochemical experiments. The mild steel specimens were mechanically cut into  $2.5 \times 2 \times 0.025$  cm (for gravimetric) and  $8 \times 1 \times 0.025$  cm (for electrochemical) dimensions and then abraded with silicon carbide (SiC) abrasive papers of grade 600, 800, 1000, and 1200, respectively, degreased in acetone, and dried at ambient temperature. The test solution, 1 M HCl, was prepared by diluting analytical grade 37% HCl with double-distilled water.

#### 2.2 Test Solution

Stock solutions of Abacavir were made by dissolving it in 1 M HCl solution and for dilution double distilled water was used.

#### 2.3 Weight loss method.

Gravimetric experiments were performed following standard method [36]. The corrosion rate  $(C_R)$  of mild steel in acidic medium was calculated by using following equation [37]:

$$C_{\rm R} = \frac{W}{At} \tag{1}$$

where, W is weight loss of mild steel specimens (mg), A is the area of the specimen (cm<sup>2</sup>) and t is the exposure time (h).

The corrosion efficiency ( $\eta$  %) [38] and surface coverage ( $\theta$ ) were calculated using following formulas.

$$\eta\% = \frac{C_{\rm R} - C_{\rm R(i)}}{C_{\rm R}} \times 100 \tag{2}$$
$$\Theta = \frac{C_{\rm R} - C_{\rm R(i)}}{C_{\rm R}} \tag{3}$$

where  $C_R$  and  $C_{R(i)}$  are the corrosion rate  $(mg \ cm^{-2} \ h^{-1})$  values in absence and presence of Abacavir respectively.

#### 2.4 Electrochemical impedance spectroscopy

Conventional three electrode assembly was used for electrochemical measurements in which mild steel with  $1\text{cm}^2$  exposed areas used as working electrode, a platinum sheet was used as the auxiliary electrode and a saturated calomel electrode (*SCE*) was used as reference electrode. The three electrode assembly connected to a Gamry Potentiostat/ Galvanostat (Model G-300) instrument. All the experiment was performed in absence and presence of optimum concentration of Abacavir in 1M HCl. The experimental data were processed by Gamry Echem Analyst 5.0 software. All the experiments were measured after immersion of mild steel for 30 min in 1 M HCl in the absence and presence of Abacavir. Prior to polarization and *EIS* experiments the electrode was allowed to corrode freely and its *OCP* was recorded as a function of time for 200 s. After this time a steady-state *OCP* corresponding to

corrosion potential ( $E_{corr}$ ) of the working electrode was obtained. The above-mentioned procedure was repeated for each concentration of the investigated inhibitor.

Polarization resistance studies were carried out from 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  $0.125 mV s^{-1}$  to study the effect of Abacavir on polarization resistance of mild steel. The polarization resistance was calculated from the slope of potential vs. current curve in the vicinity of corrosion potential. The inhibition efficiency from polarization resistance was calculated using following formula.

$$\eta\% = \frac{R_{\rm p}^{\rm i} - R_{\rm p}^{\rm o}}{R_{\rm p}^{\rm i}} \times 100 \tag{4}$$

where,  $R^{o}_{p}$  and  $R^{i}_{p}$  are the polarization resistance in absence and in presence of Abacavir, respectively.

The electrochemical polarization behaviour of mild steel specimens was study by recording the anodic and cathodic potentiodynamic curves. The potentiodynamic measurements performed in 1M HCl in absence and presence of different concentration of Abacavir by changing the electrode potential automatically from -250 to +250 mV vs. corrosion potential at a scan rate of 1 mV s<sup>-1</sup>. The corrosion current density ( $i_{corr}$ ) obtained by extrapolating the linear segments of cathodic and anodic polarization curves, from which inhibition efficiency calculated using following formula.

$$\eta\% = \frac{i_{\rm corr}^0 - i_{\rm corr}^i}{i_{\rm corr}^0} \times 100 \tag{5}$$

where  $i^{0}_{corr}$  and  $i^{i}_{corr}$  are the corrosion current density in absence and presence of Abacavir, respectively

The impedance studies were carried out using ac signals of 10 mV amplitude for the frequency range from 100 kHz to 0.01 Hz. The charge transfer resistance ( $R_{ct}$ ) values were obtained from the diameter of the semi-circles of the Nyquist plots from which inhibition efficiency calculated by using following formula.

$$\eta\% = \frac{R_{\rm ct}^{\rm i} - R_{\rm ct}^{\rm 0}}{R_{\rm ct}^{\rm i}} \times 100 \tag{6}$$

where,  $R^{o}_{ct}$  and  $R^{i}_{ct}$  are the charge transfer resistance in absence and in presence of inhibitor, respectively.

## **3. RESULTS AND DISCUSSION**

#### 3.1 Weight loss measurements

#### 3.1.1. Effect of inhibitor concentration

The values of percentage inhibition efficiency ( $\eta$ %) and corrosion rate ( $C_R$ ) obtained from weight loss measurement at different concentrations for Abacavir at 35 <sup>0</sup>C is summarized in Table 1. From table it is clear that on increasing Abacavir concentration, corrosion efficiency also increases.

Maximum efficiency was found at 400 ppm concentration further increase in concentration did not cause any appreciable change in the inhibition performance of Abacavir. From table it is also depicted that corrosion rate ( $C_R$ ) values decrease with increasing the concentration of Abacavir. The variation of inhibition efficiency and surface coverage with Abacavir concentrations is shown in Figure 2 (a, b).



Figure 2. (a) Inhibition efficiency of Abacavir at different concentrations (b) Surface coverage of Abacavir at different concentrations (c) Inhibition efficiency of Abacavir at different temperatures

#### 3.1.2. Effect of temperature:

The effect of temperature on the inhibition performance of Abacavir as corrosion inhibitor is investigated by weight loss measurements in the temperature range 308–338 K in absence and presence of Abacavir at 400 ppm concentration [figure 2(c)]. From figure, it is clear that inhibition efficiency decreases from 97 to 68 % with increasing temperature of Abacavir from 308-338<sup>0</sup>K. This decrease in inhibition is due to desorption of Abacavir from metal surface .Thus, at higher temperature , more desorption of Abacavir molecules takes place and larger surface area of metal come in contact with acid, resulting in an increase in corrosion rate [39].

**Table 1.** Corrosion rate ( $C_R$ ), Surface coverage ( $\theta$ ) and corrosion inhibition ( $\eta$ %) for mild steel in 1M HCl in absence and in presence of different concentrations of Abacavir from weight loss measurements at 308 K.

Inhibitor	Conc (ppm)	Weight loss(mg)	Surface coverage(θ)	Inhibition efficiency( <b>η%</b> )	Corrosion rate(mm/y)
Bank	0.0	219			85.3
	100	31	0.8584	85.84	11.50
Abacavir	200	19	0.9132	91.32	7.04
	300	11	0.9497	94.97	4.08
	400	5	0.9771	97.71	1.85

#### 3.1.3 Thermodynamically parameters and Adsorption isotherms:

The mechanism of corrosion inhibition may be explained on basis of adsorption behaviour [40]. Several adsorption isotherms were tested to describe the adsorption behaviour of Abacavir. The degree of surface coverage ( $\theta$ ) for different concentrations of Abacavir was evaluated from weight loss data. It is found that Abacavir obey Langmuir adsorption isotherm (Figure 3 (a)).

Corrosion rate depend upon temperature and this dependency is given be Arrhenius equation and transition state equation [41, 42].

$$C_{\rm R} = A \exp\left(\frac{-E_{\rm a}}{RT}\right)$$

$$C_{\rm R} = \frac{RT}{Nh} \exp\left(\frac{\Delta S^*}{R}\right) \exp\left(-\frac{\Delta H^*}{RT}\right)$$
(7)
(8)

where  $E_a$  is the apparent effective activation energy, R is the general gas constant, T is the temperature, k is the Arrhenius pre-exponential factor, h is the Plank's constant, N is the Avogadro's number,  $\Delta S^*$  is the entropy of activation and  $\Delta H^*$  is the enthalpy of activation. The linear plot between log  $(C_R)$  vs. 1/T and log  $(C_R/T)$  vs. 1/T were performed. As shown in Figure 3(b,c) straight line obtained with a slope  $-\Delta E_a/2.303 R$ ,  $-\Delta H^*/2.303 R$ ) and an intercept of log  $(R/Nh)+(\Delta S/2.303 R)$ ,

from which values of  $E_a$ ,  $\Delta H^*$  and  $\Delta S^*$  were calculated and given in Table 2: From table it is clear that activation energy of Abacavir is higher (55.14  $kJ mol^{-1}$ ) as compare to blank acid solution (28.48  $kJ mol^{-1}$ ) this show that corrosion rate of mild steel is mainly controlled by activation energy [43]. The negative values of  $\Delta S^*$  indicates that the process of adsorption is spontaneous [44-45]. Positive sign of  $\Box H^*$  reflected the endothermic nature of mild steel dissolution process, which suggested the slow dissolution of mild steel [46].



**Figure 3.** (a) Langmuir adsorption isotherm (b) Arrhenius plots of log  $C_R$  versus 1/T (c) Transition state plots of log  $C_R/T$  versus 1/T

Adsorption of Abacavir involves the formation of two types of interaction responsible for bonding of Abacavir to a metal surface. The first one (physical adsorption) is weak undirected interaction and is due to electrostatic attraction between inhibiting organic ions or dipoles and the electrically charged surface of metal. The second type of interaction (chemical adsorption) occurs when directed forces govern the interaction between the adsorbate and adsorbent. Chemical adsorption involves charge sharing or charge transfer from adsorbates to the metal surface atoms in order to form a coordinate type of bond. Chemical adsorption has a free energy of adsorption and activation energy higher than physical adsorption and, hence, usually it is irreversible [47].

The value of heat of adsorption was determined from the slope  $(-\Delta G_{ads}/2.303RT)$  of graph. The free energy  $(\Delta G_{ads})$  of adsorption and adsorption constant  $(K_{ads})$  were calculated by following equations:

$$\Delta G_{ads}^{\circ} = -RT \ln(55.5K_{ads})$$
(9)
$$\ln K_{ads} = \frac{-\Delta H_{ads}^{\circ}}{RT} + \text{constant}$$
(10)

The values of free energy of adsorption and adsorption constant were calculated and given in table 2. It is also seen from the Table 2 that  $K_{ads}$  decreased with increase in temperature indicating that adsorption of Abacavir on the mild steel surface was unfavorable at higher temperatures. The negative values of  $\Delta G_{ads}$ , calculated from Equation. (9) are consistent with the spontaneity of the adsorption process and the stability of the adsorbed layer on the mild steel surface. Generally, the energy values of  $-20 \ kJ \ mol^{-1}$  or less are associated with an electrostatic interaction between charged molecules and charged mild steel surface, (physisorption) those of  $-40 \ kJ \ mol^{-1}$  or more negative involve charge sharing or transfer from the inhibitor molecules to the metal surface to form a coordinate covalent bond (chemisorption) [48].

Inhibitor Blank	Ea (kJ mol <sup>-1</sup> )	-ΔG (kJ mol <sup>-1</sup> )				Kads (M-1 10 <sup>3</sup> )				$\Delta H$ (kJ mol <sup>-1</sup> )	$\frac{\Delta S}{(JK^{-1}mo^{-1})}$
		308	318	328	338	308	318	328	338		
Blank	28.48	-	-	-	-	-	-	-	-	26.04	-178.9
Abacavir	55.14	-38.30	-35.01	-34.30	-33.98	56.2	10.1	5.2	3.2	62.42	-117.4

**Table 2.** Thermodynamic parameter for mild steel in 1M HCl in absence and presence of optimum concentration of Abacavir.

#### 3.2 Electrochemical Measurements

#### 3.2.1. Electrochemical Impedance Spectroscopy

The corrosion behaviour of mild steel in 1 M HCl in absence and presence of different concentrations of Abacavir were investigated by EIS after immersion for 30 min at  $303 \pm 1$  K. Nyquist plot of mild steel in uninhibited and inhibited acid solutions containing various concentrations of Abacavir is presented in Figure. 4 (a). From figure it is clear that diameter Nyquist plot increases with

concentration. The increasing diameter of Nyquist plot in 1 M HCl in presence of Abacavir indicated the corrosion inhibition of mild steel.



**Figure 4.** (a)Nyquist plots in absence and presence of different concentrations of Abacavir (b) Equivalent circuit used to fit the impedance data

For corrosion reactions which are strictly charge transfer controlled, impedance behavior can be explained with the help of a simple and commonly used equivalent circuit [Figure 4 (b)] composed of a double layer capacitance, charge transfer resistance ( $R_{ct}$ ) and solution resistance ( $R_s$ ). The resistor  $R_s$  is in series to the double layer capacitance and  $R_{ct}$ , while double layer capacitance is parallel to  $R_{ct}$ . The double layer capacitance is in parallel with the impedance due to the charge transfer reaction. This type of circuit is used to model the iron/acid interface [49]. The *EIS* parameters for Abacavir such as  $R_s$ ,  $Y_0$ ,  $R_{ct}$  and  $C_{dl}$  were derived from the Nyquist plot are given in Table 3. It is depicted from Table, that the impedance of the inhibited system amplified with increase and the  $C_{dl}$  values decreased with increase in Abacavir concentration. The double layer capacitance ( $C_{dl}$ ) was calculated by using following equation [50]:

$$C_{dl} = Y_0 \left(\omega_{max}\right)^{n-1}$$

(11)

where,  $Y_0$  is *CPE* coefficient, *n* is *CPE* exponent (phase shift),  $\omega$  is the angular frequency. The  $\omega_{\text{max}}$  represents the frequency at which the imaginary component reaches the maximum. This decrease in  $C_{dl}$  results from a decrease in local dielectric constant and/or an increase in the thickness of the double layer, suggested that Abacavir molecules inhibit the mild steel corrosion by adsorption at the metal/acid interface [51].

400

0.986

Inhibitor	Conc (ppm)	$R_s$ ( $\Omega$ cm <sup>2</sup> )	$\begin{array}{c} R_{ct} \\ (\Omega \ cm^2) \end{array}$	$Y^0$ µF cm <sup>-2</sup> )	n	$C_{dl}$ $\mu F \text{ cm}^{-2}$ )	$\eta\%$
Blank	0.0	1.11	11.8	249.6	0.827	85.05	
	100	0.708	116.79	178.5	0.805	76.55	89.51
	200	0.723	241.97	72.17	0.851	34.93	94.94
Abacavir	300	0.857	423.74	63.24	0.855	27.78	97.11

14.3

0.838

6.37

495.31

**Table 3.** The Electrochemical Impedance parameters and corresponding efficiencies of Abacavir in 1M HCl at different concentrations

As stated earlier the diameter of Nyquist plot increases with increasing concentration of Abacavir. The decrease of diameter in Nyquist plot is a feature for solid electrodes and often referred to as frequency dispersion and attributed to the roughness and other in homogeneities of the solid electrode [52]. It is worth noting that the percentage inhibition efficiencies obtained from impedance measurements are comparable and run parallel with those obtained from weight loss and potentiodynamic polarization measurements.

## 3.2.2. Potentiodynamic polarization measurements



**Figure 5**. Tafel polarization curves for corrosion of mild steel in 1 M HCl in the absence and presence of different concentrations of Abacavir.

Potentiodynamic polarization curves of mild steel in 1 N HCl in the absence and presence of 100 to 400 ppm concentrations of Abacavir is given in Figure 5. The potentiodynamic polarization

97.52

parameters i.e. Corrosion potential ( $E_{corr}$ ), anodic and cathodic Tafel slopes ( $\beta_a$  and  $\beta_c$ ), corrosion current density ( $i_{corr}$ ) and percentage inhibition efficiency ( $\eta$  %) were also calculated and given in Table 4. It can be seen from table that  $i_{corr}$  value decreases from 212  $\mu A$  cm<sup>-2</sup> to 25.8  $\mu A$  cm<sup>-2</sup> on increasing concentration from 100 to 400 ppm this depicted that the rate of electrochemical reaction was reduced due to the formation of a barrier layer over the mild steel surface by the Abacavir molecule.

From Table 4, it is also clear that the values of cathodic and anodic Tafel slope constant are slightly changed in the presence of Abacavir. This suggest that Abacavir was first adsorbed onto the mild steel surface and impeded by merely blocking the reaction sites of the metal surface without affecting the anodic and cathodic reaction [53]. Maximum reduction of  $i_{corr}$  for each inhibitor is obtained at 400 ppm. From table, It is also observed that  $E_{corr}$  values did not change significantly in presence of Abacavir suggesting that it is a mixed type inhibitor [54].

**Table 4**. The potentiodynamic polarization and Linear polarization parameters and corresponding efficiencies of Abacavir in 1 M HCl at optimum concentration

Inhibitor	Conc (ppm)		<u>Linear</u> Polarization					
		E <sub>corr</sub> (mV vs. SCE)	i <sub>corr</sub> (μA cm <sup>-2</sup> )	$egin{array}{c} \beta_a \ (mV/dec) \end{array}$	$\beta_c$ (mV/dec)	$\eta\%$	$R_P$ ( $\Omega$ cm <sup>2</sup> )	$\eta\%$
Blank	-	-495	1320	90.0	152.0	-	11.81	-
	100	-507	212	75.6	109.4	89.0	115.4	89.76
	200	-508	71	78.7	144.3	93.54	244.2	95.16
Abacavir	300	-490	40.1	68	156.7	96.35	388.1	96.95
	400	-528	25.8	52.3	164.3	97.56	431.7	97.26

#### 3.2.3 Linear polarization method.

The inhibition efficiencies and polarization resistance  $(R_p)$  parameters are presented in Table 4. The increase in the  $R_p$  value depicted the higher efficiency as the concentration of Abacavir increases from 100 to 400 ppm. The values calculated by Tafel polarization and *EIS* data shows good agreement with the results obtained from linear polarization resistance.

#### 4. MECHANISM OF CORROSION INHIBITION

Corrosion inhibition of mild steel in hydrochloric acid solution by Abacavir can be explained on the basis of molecular adsorption. The Abacavir inhibit mild steel corrosion by controlling both anodic as well as cathodic reactions. In acidic solution, Abacavir exist as protonated species. In Abacavir the nitrogen atoms present in the molecules can be easily protonated in acidic solution and convert into quaternary compounds. These protonated species adsorbed on the cathodic sites of the mild steel and decrease the evolution of hydrogen. The adsorption on anodic site occurs through pielectrons of aromatic ring and lone pair of electrons of nitrogen atoms which decrease the anodic dissolution of mild steel [55]. A schematic illustration of different modes of adsorption of Abacavir on metal/acid interface is shown in Figure 6.



Figure 6. The schematic illustration of different modes of adsorption by Abacavir on mild steel 1 M HCl interface

The high performance of Abacavir is attributed to the presence of pi-electrons, quaternary nitrogen atom, larger molecular size and the planarity of compounds.

Thus Abacavir can adsorb on the mild steel surface by following ways:

(a) Electrostatic interaction between the charged molecules and charged metal;

(b) Interaction of  $\pi$ -electrons with the metal;

(c) Interaction of unshared pair of electrons in the molecule with the metal; and

(d) The combination of the all the effects [56-58].

# **5. CONCLUSIONS**

(1) The Abacavir is good corrosion inhibitor for mild steel corrosion in 1 M HCl solution.

(2) The Potentiodynamic polarization study revealed that Abacavir act as mixed-type inhibitor.

(3) The inhibition efficiency order of Abacavir increases with increase in concentration and maximum efficiency (98%) was observed at 400 ppm concentration.

(4) The adsorption of Abacavir on mild steel surface obeys the Langmuir adsorption isotherm.

(5) The inhibiting efficiencies obtained by polarization, *EIS* and weight loss measurements are in good agreement.

(6) The negative sign of the  $\Delta G_{ads}$  indicates that the adsorption of Abacavir on the mild steel surface in 1 M HCl is spontaneous process.

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