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Investigating the Anticorrosion Potentials of Expired Nevirapine Antiretroviral as Inhibitor of Potential Crude Oil Steel in Acidic Medium

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***Abstract* - The anticorrosion potential of expired nevirapine antiretroviral drug was investigated using both chemical (gravimetric and thermometric) and electrochemical (electrochemical impedance spectroscopy and potentiodynamic polarization) techniques. In both methods of measurements, the nevirapine inhibitor acted as an excellent inhibitor (98.9% inhibition efficiency). The inhibitor was good at all the tested temperatures but with best active performance at lower temperatures (98.1 %, 89.8 % and 84.6 % at 313 K, 323 K and 333 respectively), which explains a physical adsorption process. There was decreasing corrosion rate as inhibitor concentration was increased indicating that the nevirapine significantly halted the 0.5 M HCL acid reaction on the steel. The analysis showed that the charge transfer resistance values increased while the double layer capacitance values decreased with increasing nevirapine concentration which by this confirmed the earlier results from chemical techniques commending the excellent performance of the inhibitor. Langmuir adsorption showed good correlation and a monolayer adsorption process while the absorption free energy described the inhibitor as being stable and spontaneous.**

Keywords: polarization, Langmuir, enthalpy, inhibition, thermometric, nevirapine, impedance.

I. INTRODUCTION

Metallic materials constitute the most widely used group of materials particularly in both mechanical and the transportation industry. They are also used in electronics and increasingly in the construction industry. Mild steel is the most commonly used engineering material. It is cheap, readily available in a wide range of standard forms and sizes, and can easily be worked upon and welded. It has good tensile strength and ductility. However, the usefulness of mild steel, and indeed, metals and alloys; is constrained by one common challenge known as corrosion. Corrosion is a naturally occurring phenomenon simply defined as deterioration of metal surfaces caused by reaction with the surrounding environmental conditions [1]. Acid solutions (basic ones in some cases), commonly used in the chemical industry to remove mill scales from metallic surfaces; constitute one of such environments that breed corrosion. Hydrochloric acid is widely used in removing scales and salts from steel surfaces, cleaning tanks and pipelines and steel alloys. Corrosion can cause disastrous damage to metal and alloy structures; causing economic consequences in terms of repair, replacement, product losses, safety and environmental pollution. Due to these harmful effects, corrosion is an undesirable phenomenon that ought to be prevented. Corrosion problems occur in the petroleum industry in at least three general areas: (1) production, (2) transportation and storage, and (3) refinery operations [2].

Several ways of preventing corrosion and the rates at which it can propagate with a view of improving the lifetime of metallic and alloy materials are in practice. The use of inhibitors for the control of metals and alloys in contact with aggressive environment is one among the acceptable practices used to reduce and/or prevent corrosion. A corrosion inhibitor is a substance which when added in small concentration to an environment, effectively reduces the corrosion rate of a metal exposed to that environment. The inhibitors can be broadly classified into two categories namely those that enhance the formation of a protective oxide film through an oxidizing effect and those that inhibit corrosion by selectively adsorbing on the metal surface and creating a barrier that prevent access of corrosive agents to the metal surface [2,3].Several organic compounds have been studied and analyzed to investigate their potential as corrosion inhibitors. Most of these studies reveal that almost all organic molecules containing heteroatoms such as nitrogen, sulphur, phosphorous and oxygen show significant inhibition efficiency. Despite these

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promising findings about possible corrosion inhibitors, most of these substances are not only expensive but also toxic and non-biodegradable; thus, raising pollution issues [2, 4]. These deficiencies have prompted the search for their replacements. Several researches have proven that antibiotics can be used as potential corrosion inhibitors, because through their functional groups, they form metal ions on metal surfaces. Antibiotics fall under the category of eco friendly compounds and hence claimed as Green corrosion inhibitors which are completely soluble in aqueous media and obtainable with high purity and relatively cheap. These properties will validate their use as corrosion inhibitors in various media. It has the efficacy to combat the corrosion of a wide variety of metals such as mild steel, stainless steel aluminum, zinc and nickel. Several techniques have been used to evaluate the corrosion inhibition performances of antibiotics and to analyze the nature of protective film formed on the metal surface.

Nevirapine is a benzodiazepine non-nucleoside reverse transcriptase inhibitor used in combination with other agents in the therapy of human immunodeficiency virus infection and the acquired immune deficiency syndrome. Nevirapine has a molar mass of 266.3 g.

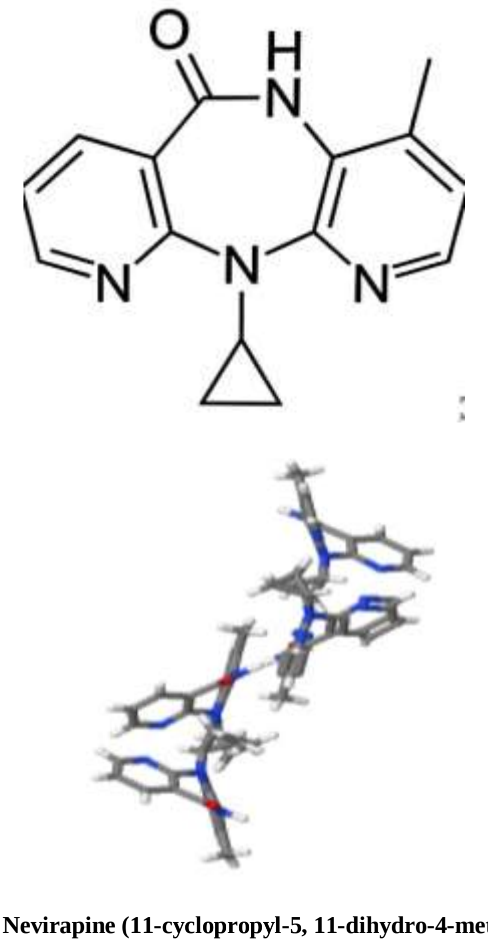

Figure 1: Nevirapine (11-cyclopropyl-5, 11-dihydro-4-methyl-6H-dipyrido (3,2-b:2,3-e)(1,4)diazepin-6-one) $C_{15}H_{14}N_4O$

II. MATERIALS AND METHODS

a) Gravimetric experimentation

The potential crude oil steel cut into dimension of 3.5 x 0.08 x 5 cm was mechanically polished with silicon carbide abrasive paper, degreased with ethanol, washed with deionized water, rinsed with acetone and air dried. Each of the resized steel was weighed and suspended in a 100 mL glass beaker containing 100 mL of 0.5 M HCl solution. The corrosion vessels were charged with different concentrations (1.0 x 10^{-4}, 2.0 x 10^{-4} M, 3.0 x 10^{-4} M, 4.0 x 10^{-4} M, 5.0 x 10^{-4}M) of the Nevirapine inhibitor, allowed to stand for 60 minutes and covered with paraffin to prevent contamination and evaporation of solution. The steel sheets were retrieved, dipped in distilled water and scrubbed with bristle brush to remove residual acids. They were washed thoroughly (and rinsed) with distilled water, rinsed in acetone, air dried and reweighed. Triplicate measurements were made. This procedure was repeated at different concentrations progressively for 5 hr.

b) Thermometric Experimentation

Hundred ml of the 0.5 M HCl solution was released into a round bottom flask and the potential crude oil steel of dimension 3.5 cm x 0.08 cm already weighed was dropped into the beaker while the beaker was immediately immersed in a water bath. The volume of the hydrogen gas evolved from the corrosion reaction was regulated by temperature changes in the water bath. After every 1 hour at different temperatures of 313 K, 323 K and 333 K, the metal was removed, degreased with ethanol, rinsed with acetone and air dried and then weighed. This procedure was repeated for a set of fresh metals at different concentrations of 1.0 x 10^{-4}, 2.0 x 10^{-4} M, 3.0 x 10^{-4} M, 4.0 x 10^{-4} M, 5.0 x 10^{-4} M for the inhibitors.

c) Electrochemical Experimentation

The steel of dimension 1 cm x 1 cm, graphite rod and silver/silver chloride (Ag/AgCl) were used as working, counter and reference electrodes, respectively for the Potentiodynamic polarization measurement using the Gamry electrochemical instrument framework. The potentiodynamic polarization curves were recorded from cathodic potential of -2.0 eV to anodic potential of +2.5 eV at a scan rate of 0.5 mV s^{-1} with respect to free corrosion potential (E_{corr}). The linear Tafel segments of the anodic and cathodic curves were extrapolated to corrosion potential to obtain the corrosion current densities (i_{corr}) and other electrochemical parameters of interest.

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III. RESULTS AND DISCUSSIONS

a) Effect of inhibitor concentration on corrosion rate (Gravimetric analysis)

The weight loss data was used to calculate the corrosion rate (R) in mg (equation 1), percentage inhibition efficiency ($\%IE$) (equation 2) and degree of surface coverage (θ) (equation 3)in accordance with the study of [2]:

$$R = \frac{\Delta w}{At}\ x\ 1000 \quad (1)$$

Where Δw = average weight loss, A = exposed surface area of the steel sheet, and t = time of immersion in the solution.

$$\%IE = \frac{\nu_o - \nu_i}{\nu_o} \times 100 \quad (2)$$

Where ν_o = corrosion rate in absence of inhibitor, ν_i= corrosion rate in presence of inhibitor.

$$\theta = \frac{\%IE}{100} \quad (3)$$

A clear concentration dependence of the corrosion rate and, consequently, inhibitor efficiency and fractional surface coverage, was observed as shown in Figures 1 and Table 1.

The corrosion rate decreased to a minimum value at Nevirapine concentration of 5.0 x 10^{-4} in the corrosion medium. This change in corrosion rate had a very strong impact on the inhibition efficiency, which increased with increasing concentration of the inhibitor to reach a maximum value of 98.9 %. This proves Nevirapine as an effective green inhibitor for potential crude oil steel [5-8]. Similar impact was observed for fractional surface coverage.

According to [9], the observed trends (Table 1) are so because increasing concentration of the inhibitor reduces the steel surface area exposed to the corrosion media through the progressive increase in number of adsorbed inhibitor molecules on its surface, which hinders direct attack of the corrosion agent on the metal surface [8,9,10].

TABLE 1

Results from corrosion rate of metal, surface coverage and Nevirapine inhibitor concentration in acidic medium calculated from gravimetric experiment

Conc. (M)	CR (mg/cm2/h)	θ	%IE
0.5 M HCl	0.957	-	-
1.0 x 10^{-4} M	0.298	0.689	68.9
2.0 x 10^{-4} M	0.247	0.742	74.2
3.0 x 10^{-4} M	0.172	0.820	82.0
4.0 x 10^{-4} M	0.101	0.894	89.4
5.0 x 10^{-4} M	0.011	0.989	98.9

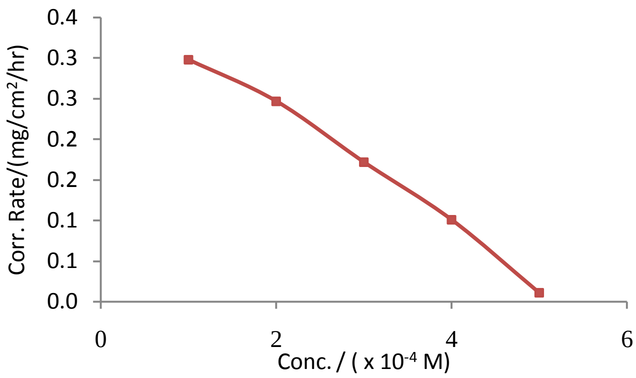

Figure 2: Plot showing the relationship between corrosion rate of steel and inhibitor concentration in 0.5 M HCl solution

b) Effect of temperature on Nevirapine inhibitor (Thermometric analysis)

The results obtained however showed that the corrosion rate decrease with increase in concentrations of both inhibitors, but increased with increase in temperature (Table 2). From the corrosion rates, the inhibition efficiency was calculated and the result showed increases %IE with increase in inhibitor concentrations as well as decrease with increase temperature. This revealed that, the inhibitor adsorbed on the metal surface thereby protecting metal from the action of acid and the latter suggest a desorption phenomenon following weak forces of attraction due to high temperature agitation effect [10-13]This is in agreement with the observation of other researchers. The trend in temperature suggests physical adsorption respectively.

TABLE 2

Results from corrosion rate of metal, surface coverage and Nevirapine inhibitor concentration in acidic medium calculated from gasometric experiment

	CR (mg/cm2/h)			θ			% IE		
Conc. (M)	313K	323K	333K	313K	323K	333K	313K	323K	333K
0.5 M HCl	2.199	5.120	10.133	-	-	-	-	-	-
1.0 x 10^{-4}	1.011	2.564	5.173	0.540	0.499	0.489	54.0	49.9	48.9
2.0 x 10^{-4}	0.719	2.271	4.819	0.673	0.556	0.524	67.3	55.6	52.4
3.0 x 10^{-4}	0.397	1.382	3.101	0.819	0.730	0.694	81.9	73.0	69.4
4.0 x 10^{-4}	0.314	0.823	2.794	0.857	0.839	0.724	85.7	83.9	72.4
5.0 x 10^{-4}	0.042	0.524	1.565	0.981	0.898	0.846	98.1	89.8	84.6

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Volume 3, Issue 4, pp 44-50, April-2019

c) Effect of inhibitor on charge transfer resistance and double layer capacitance (Nyquist analysis)

Figs. 2 isa representative Nyquist plot obtained from the corrosion inhibition of Expired Nevirapine Antiretroviral drugs (ENAD) on steel in 0.5 M HCl environment. It is a perfect semicircle and this could be attributed to charge transfer reaction [11, 12]. The impedance readings for the various impedance parameters derived from Nyquist plot are tabulated in Table 3. It is observed that the double layer capacitance showed decrease as ENAD concentration increases. This could be consequence upon an increase in thickness of electrical double layer further suggesting that the inhibitor molecules function by adsorption at the metal-solution interface [7,13,14]. The values of the charge transfer resistance are seen to be increasing with increasing concentration of ENAD suggesting the strong adsorption of the inhibitor on the metal surface and decrease in further oxidation of the steel at the anodic site [13, 15].

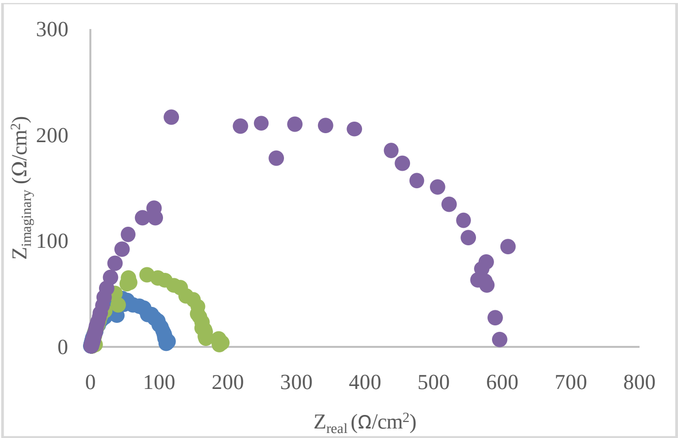

Figure 3: Nyquist Plot showing the corrosion inhibition of steel in 0.5 M HCl solution

d) Effect of inhibitor on corrosion current density and tafel slopes (Tafel analysis)

The anodic and cathodic polarization curves of steel in 0.5 M HCl with and without the various concentrations of ENAD is shown in Figure 4. The Potentiodynamic polarization parameters (corrosion current density I_{corr}, anodic and cathodic slopes, βa and βc respectively) are shown in Table 3. Table 3 show that both the cathodic and anodic curves had lower current density values in the presence of the inhibitor than those recorded in the free 0.5 M HCl solution. This suggested a reduction in the rate of dissolution of the active sites (anodic part) on the steel in 0.5 M HCl as well as the cathodic hydrogen ion reduction [12, 14]. The polarization curves in the presence of inhibitor showed similar cathodic and anodic features as the free solution suggesting that the inhibitors do not change the mechanism of the cathodic hydrogen gas evolution associated with the corrosion process [15, 16].Values of the inhibition efficiency of the inhibitor revealed a stronger adsorption tendency on the steel and this is in accordance with the result obtained from the thermometric analysis.

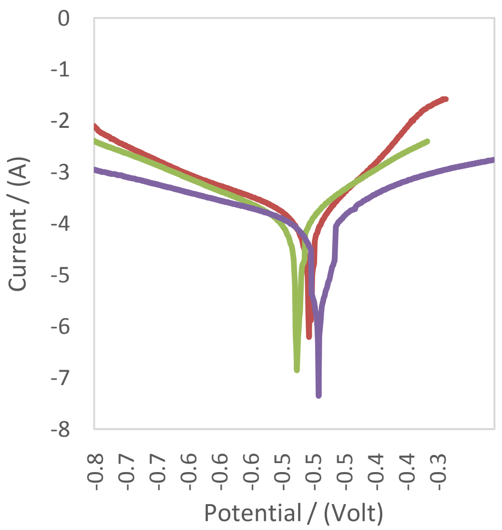

Figure 4: Tafel plot showing the corrosion inhibition of steel in 0.5 M HCl solution

TABLE 3

Results from Nyquest and Tafel plots of Nevirapine inhibitor with mild steel in acidic medium

Conc. (g/L)	Icorr (mAcm⁻²)	βc (mV/dec)	βa (mV/dec)	IEi (%)	Rct (Ωcm⁻²)	Cdl (μFcm⁻²)	% IER
0.5 M HCl	33.097	92	119	-	103	1.04	-
1.0 x 10^{-4} M	2.013	57	71	93.9	233	0.73	55.8
5.0 x 10^{-4} M	0.301	22	35	99.1	621	0.18	83.4

e) Thermodynamic treatment

The adsorption equilibrium constant (K_{ads}) for the corrosion process calculated from the Langmuir isotherm is related to the standard free energy of adsorption (ΔG^{o}_{ads}) by the equation 4,

$$\Delta G^{o}_{ads} = -2.303RT\log(55.5K_{ads}) \qquad (4)$$

Where R is the gas constant, T is the thermodynamic temperature and 55.5 is the molar concentration of water in the solution.

Activation energy (E_a) of the process was evaluated using modified Arrhenius equation (Equation 5) to relate corrosion rate and temperature,

$$\log\left(\frac{r_2}{r_1}\right) = \frac{E_a}{2.303R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \qquad (5)$$

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Volume 3, Issue 4, pp 44-50, April-2019

Where r_1 and r_2 are the corrosion rates at two temperatures T_1 and T_2 respectively and R is the gas constant.

From Table 2, the measure of the efficiency of an inhibitor has best values of 98.1 %, 89.8 % and 84.6 % at 313 K, 323 K and 333 respectively. This shows that the inhibition efficiency of the ENAD decreases with increase in temperature. It's been noted that for an inhibitor whose inhibition efficiency decreases with increase in temperature, the value of the activation energy (E_a) for the process would be greater than that for the uninhibited process.

Figure 5and Table 4 presents the activation energy profile for this process; which shows that the activation energy for the inhibited process is greater than that for the uninhibited process and decreases with increase in concentration of the inhibitor to fairly steady at a value still higher than that of the uninhibited process. Higher values of activation energy (in the presence of inhibitor) suggest that physisorption (physical adsorption) dominates in this process [13]. This is evident by the standard free energy of adsorption (ΔG^o_{ads}).

The ΔG^o_{ads} values, as presented in Table 5, were all negative for the process at both ambient and elevated temperature. This suggests spontaneity of the adsorption process and stability of the adsorbed layer on the steel surface [9,12,18]. It has been reported [20] that physisorption processes usually have $\Delta G^o_{ads} <$ -40 kJmol^{-1}, and above this value, sorption is of the chemisorption mechanism. This suggests, therefore, that the adsorption process here was physical (i.e. physisorption) since the ΔG^o_{ads} values are less than -20.0 kJmol^{-1} [5, 9].Values of ΔH_{ads} obtained from the plots are presented in Tables 4. The results obtained showed positive values of enthalpy of adsorption, an indication that the inhibition reaction is endothermic.

Also, the inhibitor is physically adsorbed on the metal surface being that the values are within the threshold of physical adsorption (< 80kJmol^{-1}).It was also noticed that the values were decreasing with increase niverapine concentration which suggested that the bond formation was increasing following increased concentration, since bond formation always follow decreased heat content [19].

However, the values of entropy of adsorption were negative indicating the absence of disorderliness, giving rise to rather association of the inhibitor molecules [10,17].

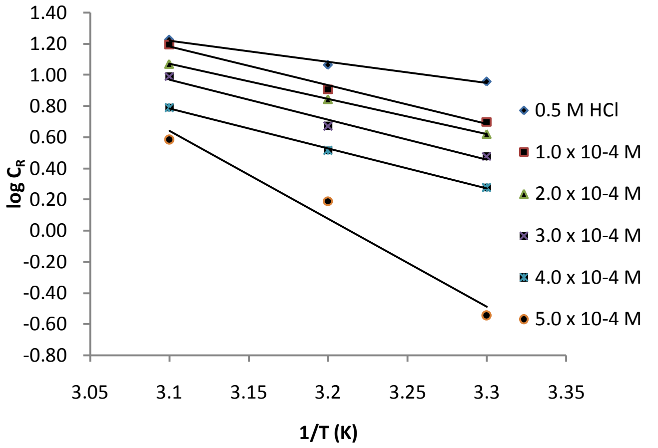

Figure 5: Plot showing the relationship between log CR and reciprocal of Temperature (K) for corrosion of steel in 0.5 M HCl solution

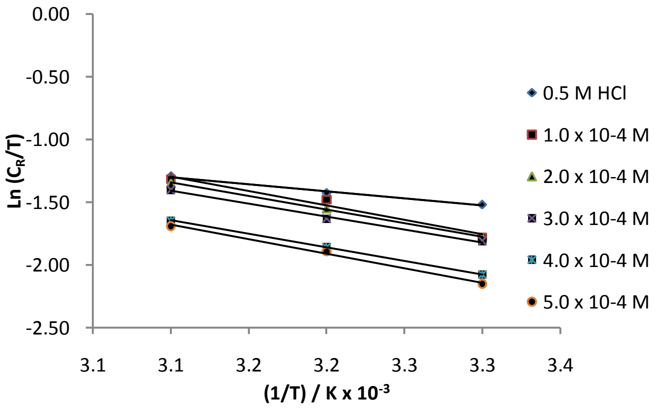

Figure 6: Plot showing the relationship between log CR and reciprocal of Temperature (K) for corrosion of steel in 0.5 M HCl solution

TABLE 4
Thermodynamic parameters describing adsorption of Nevirapine on steel in 0.5 M HCl solution

Conc. (g/L)	Ea	ΔH_{ads} kJ/mol	ΔS_{ads} kJ/mol
0.5 M HCl	10.11	27.46	-128.09
1.0×10^{-4} M	15.73	25.29	-166.15
2.0×10^{-4} M	16.47	21.58	-173.07
3.0×10^{-4} M	16.92	19.46	-188.34
4.0×10^{-4} M	17.16	18.22	-206.85
5.0×10^{-4} M	19.68	14.64	-218.30

f) Adsorption investigation

An adsorption study was investigated using the surface coverage at different concentrations to fit curves for Langmuir isotherm [20] in its linear form (Equation 6).

ISSN (online): 2581-3048

Volume 3, Issue 4, pp 44-50, April-2019

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad (6)$$

Where C is the concentration of the ENAD inhibitor in the corrosion medium and K_{ads} is the adsorption equilibrium constant. A plot of C/θ against C, which represents the Langmuir isotherm, showed linearity (Figure 7) with a very strong correlation coefficient (R^2) equal to 0.99 and slope equal to 1.061 for the process at ambient temperature (Table 5). The strongly correlated linearity suggests adherence of the experimental data for ENAD to the Langmuir adsorption isotherm [20].

TABLE 5
Langmuir Adsorption parameters describing adsorption of Nevirapine on steel in 0.5 M HCl solution

Temp. (K)	k (g/L)	R^2	Slope	ΔG^*_{ads} (kJ/mol)
313	2.141	0.997	1.102	-12.431
323	2.475	0.993	1.025	-12.810
333	3.374	0.981	0.068	-13.616

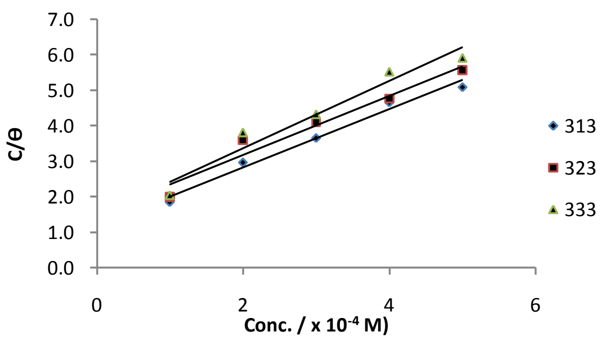

Figure 7: Langmuir isotherm plot showing the relationship between C/Ө and Concentration (M) for corrosion of steel in 0.5 M HCl solution

IV. CONCLUSION

The corrosion rate decreased to a minimum value at Nevirapine concentration of 5.0 x 10^{-4} in the corrosion medium. This change in corrosion rate had a very strong impact on the inhibition efficiency, which increased with increasing concentration of the inhibitor to reach a maximum value of 98.9 %.Hence, crude oil production, transportation and storage, and refinery operations can conveniently be carried out in the presence of ENAD as inhibitor.

The inhibitor decreased the charge transfer resistance and double layer capacitance of the metal suggesting an increase in thickness of electrical double layer, and further suggesting that the inhibitor molecules function by adsorption at the metal-solution interface.

It was observed that the effect of the nevirapine inhibitor on the steel reduced both the cathodic hydrogen evolution and anodic dissolution of the metal, suggesting a mixed type inhibition.

Thermodynamic parameters suggest spontaneity of the adsorption process and stability of the adsorbed layer on the steel surface. Also, that physisorption (physical adsorption) dominates in this inhibition process.

There is a strongly correlated linearity observed suggesting adherence of the experimental data to the Langmuir adsorption isotherm and a monolayer adsorption of nevirapine.

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Benedict Ugiis currently a Researcher and a Physical Chemistry Lecturer in the Department of Pure and Applied Chemistry of the University of Calabar, Nigeria. He obtained a Bachelor of Science (B.Sc.) Degree in Applied Chemistry from the Department of Pure & Applied Chemistry, University of Calabar, Nigeria in 2005. He proceeded to acquire Master of Science (M.Sc.) and Doctor of Philosophy (Ph.D) Degrees in 2012 and 2015 respectively both in Physical Chemistry from same University. He has served as a Lecturer with the Cross River State University of Technology, Calabar – Nigeria from 2016 - 2018. He has also served in a number of Committees in both Departmental and Faculty levels. He is a member of some academic bodies including Chemical Society of Nigeria and has published in both reputable National and internationally journals in areas of Surface Chemistry, Corrosion Science, Material Science and Electrochemistry.

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