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Assessment of Anti-Corrosion Potential of *Datura Metel* Seed Extract on Mild Steel in Acidic Medium

Catherine Feyisayo Ajayi[1], Emmanuel Folorunso Olasehinde[2], Daniel Babatunde Lene[3]
[1-3](Department of Chemistry Federal University of Technology Akure, Nigeria)

Abstract
The corrosion inhibitive behaviour of Datura metel seed extract on mild steel in acidic environment was investigated using gravimetric, electrochemical and spectroscopic methods. The results suggest that Datura metel seed extract acts as an efficient eco-friendly corrosion inhibitor for mild steel in 1 M HCl solution. The inhibition efficiency increased with an increase in inhibitor concentration and temperature, respectively. Maximum corrosion inhibition efficiency was found to be 95% at 0.5 g/mL and 333 K. Thermodynamic parameters such as E_a, ΔH^o, ΔS^o *and* ΔG°_{ads} *were also evaluated. The results revealed that corrosion inhibition may be due to the spontaneous chemical adsorption of the plant constituents on the surface of mild steel. The adsorption of the extract on the mild steel surface was found to obey Langmuir's adsorption isotherm. Atomic Absorption Spectroscopy (AAS) analysis confirmed that the dissolution of iron (*Fe^{2+}*) in the uninhibited solution was faster than the inhibited solution. The mechanism of inhibition and formation of Fe-inhibitor complex was confirmed by FT-IR spectral analysis. The results showed that the inhibition of corrosion of mild steel occurs through adsorption of the inhibitor molecules. The scanning electron microscopy (SEM/EDX) result revealed the formation of a protective layer on the mild steel. Potentiodynamic polarization curves indicated that the plant extracts behave as mixed-type inhibitor. Preliminary phytochemical analysis of the extract revealed the presence of alkaloids, phenols, flavonoids and glycosides.*

Keywords: *Mild steel, Datura metel seed, Inhibition, Adsorption, Electrochemical method*

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I. Introduction

Acid is one of the solutions often used in industries in specific applications such as acid pickling of steel, chemical cleaning and processing, ore production and oil well acidizing [1]. Steel is one of the most common materials globally, and a major component in building, infrastructure, pipeline transport, offshore construction tools, ships, automobiles and machines. The corrosion resistance of steel is poor, especially mild steel because it rusts easily, and will not be economical for usage in a corrosive environment unless some form of corrosion protection is employed [2]. It has prompted researchers worldwide to profer various solutions to improve the lifespan of metallic and alloy materials [3]. Previous investigators have shown that synthetic inhibitors are effective in the mitigation of corrosion of mild steel in acidic media. Inhibitors such as chromates and nitrite are well documented to have as much as 99% inhibition efficiency. Large numbers of organic compounds have been studied and analysed to investigate their potential as corrosion inhibitors [4]. Most of these studies revealed that almost all organic molecules containing heteroatoms such as nitrogen, sulphur, phosphorous and oxygen show significant inhibition efficiency. Despite these promising findings about possible corrosion inhibitors, most of these substances are expensive and toxic and non-biodegradable, thus detrimental to the environment [3, 5]. Hence, investigations are focused on the development of naturally occurring substances as ecofriendly corrosion inhibitors. Due to bio-degradability, eco-friendliness, low cost and easy availability, the extracts of some common plants based chemicals and their by-products have been documented as inhibitors for metals under different environments [6-11]

However, due to the vast varieties of plants, only relatively few have been thoroughly investigated. Thus, there is a dearth of information on the corrosion inhibitory potential of *Datura metel* seed extract on mild steel in acidic medium.

Datura is a genus of nine species of poisonous vespertine flowering plants belonging to the family of solanace. It is primarily an annual herbaceous plant, though occasionally biannual. Reports have shown that it originated from northern India, and is now found throughout southeast Asia. It also spread to Africa and Central and South America, and the Caribbean through human migration [12,13]. *Datura metel* seed extract is rich in flavonoids, glycosides, alkaloids and some other phytochemicals. Eddy and Ebenso 2008 reported that sapnins, tannins and alkaloids are active constituents of most green inhibitors; therefore the potency of *Datura metel* seed as corrosion inhibitor is plausible due to the presence of these active constituents [14]. However, the literature survey revealed that no study had been conducted on the inhibitive properties of *Datura metel* seed on mild steel in acidic medium. Therefore, the study investigated the inhibitory and adsorptive effects of *Datura metel* seed on mild steel in acidic medium using gravimetric, electrochemical and spectroscopic methods.

II. Materials And Methods

2.1 Materials

All chemical reagents used were of analytical grade. The materials used for this study include; Mild steel, *Datura metel* seeds, hydrochloric acid, distilled water, ethanol, talcum powder, thermostated water-bath, vernier caliper, thong, emery paper, thermometer, 4-digit analytical balance (OPD-E104 model), funnel, clean handkerchief, paper tape, Whatman filter paper and glass wares.

2.2 Preparation of the Specimen

Mild steel specimen with the following composition (wt.%): 0.12% C, 0.17% Si, 0.56% Mn, 0.02% P, 0.03% S, 0.11% Ni, 0.08% Cr and bal. Fe were used for this study. The mild steel was pressed cut into coupons of dimensions 18 x 15 x 5 mm at the Department of Material and Metallurgical Engineering, School of Engineering and Engineering Technology, Federal University of Technology Akure, Nigeria. Each coupon was polished mechanically using SiC emery papers, washed thoroughly with distilled water and degreased with ethanol and acetone, air dried in a desiccator. The weight of each metal sample before and after immersion into the corroding media was taken using 4-digit OPD-E104 analytical balance. Also, the dimensions of each coupon were noted using digital vernier caliper (0-150 mm).

The weight of the coupon after experimental period is given as:

$$\Delta W = W_1 - W_2 \qquad 3.1$$

Where W_1 *and* W_2 are the weights of the metal sample before and after exposure to the corrosive medium in the absence and presence of the plant extract.

2.3 Collection and Preparation of the Seed Extract

The *Datura metel* seeds used for this research were obtained from a site in Akure, Nigeria.

The seeds were air-dried to constant weight and ground into fine powder by an electrical blender in order to facilitate maximum effective contact of the solvent. The pulverized sample was sieved with a sieve 1 mm of moderate mesh size and later stored in a clean, dry and sealed container for the chemical analysis. Ethanol extracts of the powdered sample was obtained by standard method [15]. The extract was then filtered first through a mesh (850 micron meter sieve) and finally with Whatman number 1 filter paper. The filtrate was further subjected to evaporation over a boiling water bath and finally, a deep-brown solid residue was obtained and preserved in a desiccator. The extract was then screened both qualitatively and quantitatively to determine its phytochemical constituents. The corrosion test solutions were prepared by dissolving specified amount of the extract in 1 M HCl solution and used for corrosion study.

2.4 Corrosion Inhibition Studies

2.4.1 Gravimetric experiment

2.4.1.1 Concentration studies

The concentration of the extract was varied from 0.1 g/mL to 0.5 g/mL. Each coupon of known dimension and initial weight (W_1) was immersed in 100 mL of 1 M HCl solution in the absence and presence of the different concentrations of the plant extract for 4 hours after which it was retrieved, washed with distilled water and ethanol, dried and re-weighed (W_2). The weight of the coupons before and after immersion was calculated and taken as the weight loss and from the weight loss data, the corrosion rate (C_R) and the inhibition efficiency (I.E) of the extract at different concentrations were calculated using the equations below:

$$C_R = \frac{\Delta W}{AT} \;\; (gh^{-1}cm^{-2}) \qquad 3.2$$

Where C_R is the corrosion rate, Δw is the weight difference (loss) i.e. the final weight – initial weight, A is the area of the coupon in cm^2 and t is the time in hour.

$$I.E = \frac{C_{R(blank)} - C_{R(inhibitor)}}{C_{R(blank)}} \times 100 \qquad 3.3$$

2.4.1.2 Temperature variation studies

The effect of temperature on the corrosion rate of the steel coupons in a 100 mL 1 M HCl solution at 303, 313, 323, 333 K was also studied with the same concentration of the extract for immersion periods of 3 hours in a thermostated water bath [9]. The pre-weighed coupons were immersed in blank solution and varied with the concentrations of the inhibitor. After 3 hours, the steel coupons were retrieved, washed with distilled water and ethanol, dried at room temperature and re-weighed. The results obtained were fitted into different isotherms and the thermodynamic parameters were calculated.

2.4.2 Electrochemical study

The potentiodynamic polarization and open circuit potential (OCP) measurement experiments were carried out using VERSTAT 4 instrument. The electrochemical studies were carried out using a three-electrode cell assembly at room temperature [16]. The mild steel of geometric area of 0.786 cm^2 embedded in resins was used as the working electrode platinum electrode was used as counter electrode and saturated silver/silver chloride was used as reference electrode while 1 M solution of HCl in the presence and absence of the extract serves as the electrolyte. The working electrode was polished with different grades of emery papers. Prior to the potentiodynamic polarization measurement, the open-circuit corrosion potential (OCP) measurements were carried out from cathodic potential of -250 mV to an anodic potential of +250 mV with a scan rate 1.0 mVs^{-1} for 30 minutes to determine the current density.

2.4.3 Fourier Transform-Infrared Measurement

Fourier transform-infrared spectrophotometer (FT-IR) has been established as a powerful instrument that can be used to determine the type of bonding for organic inhibitors absorbed on the metal surface [17]. The steel was pulverized into fine particles and dissolved in the solution of hydrochloric acid containing extract for 4 hours to form the adsorption product. The solution was filtered after the immersion and the residue was dried into powdery form which was used for FT-IR analysis together with the extract that was not dissolved in acid. FT-IR spectra of both the extract and the adsorption product were obtained in KBr with a Fourier Transform Spectrometer (Shimadzu model 8400S) for the detection of various functional groups present in the inhibitor and values are given in cm^{-1}.

2.4.4 Atomic Absorption Spectroscopy (AAS) Analysis

Atomic absorption analysis was conducted by using Atomic Absorption Spectrometer model Buck Scientific 210. The coupons were immersed in 100 mL of 1 M HCl with and without various weights of the inhibitor. After 4 hours of immersion, the coupons were removed and the solutions were filtered and the filtrates were used for the analysis. This was done in order to determine the amount of iron (II) ions dissolved in both inhibited and uninhibited solution after 4 hours of immersion. The calibration curve of iron (II) ions was drawn before analyzing the electrolyte solution. All samples containing iron (II) were diluted with distilled water to ensure that the concentration of the metal ions is within the range of the calibration curve [6].

2.4.5 Scanning Electron Microscopy (SEM/EDX) Analysis

Analysis of mild steel surface and the formation of a passive layer on the surface with and without inhibitor was studied using Scanning Electron Microscopy [19]. The surface morphologies with the elemental composition of the mild steel used for this study were assessed by Scanning Electron Microscope/Energy Dispersive X-ray Spectrometer (Nikon Eclipse ME600). To perform this test, mild steel specimens were immersed in 1 M HCl in the presence and absence of inhibitor for 4 hours at room temperature. To observe the effect of inhibitor on the metal, only 0.5% g/mL of the inhibitor which gives the maximum efficiency was considered and the surface morphology of the metal in the inhibited solution was compared with that of uninhibited solution.

III. Results and Discussion

3.1 Phytochemical Screening

The phytochemical constituents of extract of *Datura metel* seed was determined qualitatively and quantitatively and the results are presented in Table 1.

The results revealed the presence of saponins, tannins, flavonoid, phytates, phenols, oxalates, phytic acids, glycosides and alkaloids while anthraquinones and terpenoids were absent in the extract. Previous studies have shown that most of these phyto-constituents containing electron rich bonds or heteroatoms that facilitate their electron donating ability to the vacant d-orbitals of the metal may be responsible for the inhibition process [14, 20, 21]. Thus, the inhibitory potential of ethanol extract of *Datura metel* seed on mild steel can be attributed to the presence of these phyto-chemicals.

Table 1 Phyto-constituents of *Datura metel* seed extract

Phytochemicals	Qualitative	Quantitative
Saponins (%)	+	3.561
Tannins (mg/g)	+	0.656
Flavonoids (%)	++	14.543
Phytates (mg/g)	++	12.360
Phenols (%)	++	16.375
Oxalates (mg/g)	+	4.052
Phytic acids (mg/g)	+	3.482
Glycosides (mg/g)	+++	43.853
Alkaloids (%)	++	17.312

+ = Present
++ = Adequately present
+++ = Highly present

3.2.1 Effect of extract concentrations on corrosion rate of mild steel

The weight loss technique was used for the study of the inhibitive effect of *Datura metel* seed extract under different concentrations. The variations of corrosion rate of mild steel in 1M HCl in the absence and presence of various concentrations of the extract of *Datura metel* seed are shown in Figure 1. The results showed that the corrosion rate of the mild steel in 1M HCl decreases with increase in concentration of the extract, indicating that the rate of corrosion of mild steel in acid is dependent on the amount of inhibitor present [22, 23]. The decrease observed in the weight loss with increase in the concentration of the extract suggests an increased surface coverage on the metal and adsorption of phyto-constituents on the metal surface creating a barrier between the metal and the acidic medium [21]. Therefore, based on this observation, it can be concluded that the ethanol extract of *Datura metel* seed effectively retarded the corrosion of mild steel in acidic medium.

3.2.2 Effect of extract concentrations on inhibition efficiency of mild steel

As shown in Figure 2, the inhibition efficiency of the extract on mild steel was observed to increase with increase in concentration of the extract. As the concentration of the inhibitor increases from 0.1 g/mL to 0.5 g/mL, percentage inhibition efficiency increases from 66% to 95 %. The increase is due to the increase in the number of constituent molecules of *Datura metel* seed adsorbed on the metal surface at higher concentrations protecting the active

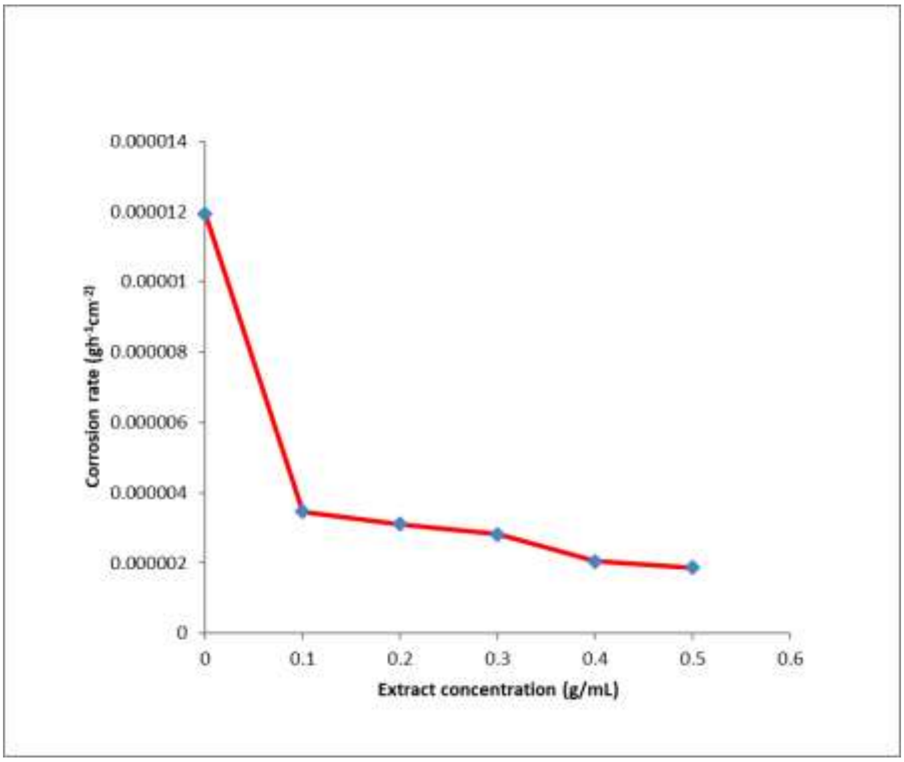

Figure 1: Effect of concentration of *Datura metel* seed extract on the corrosion rate of mild steel in 1 M HCl

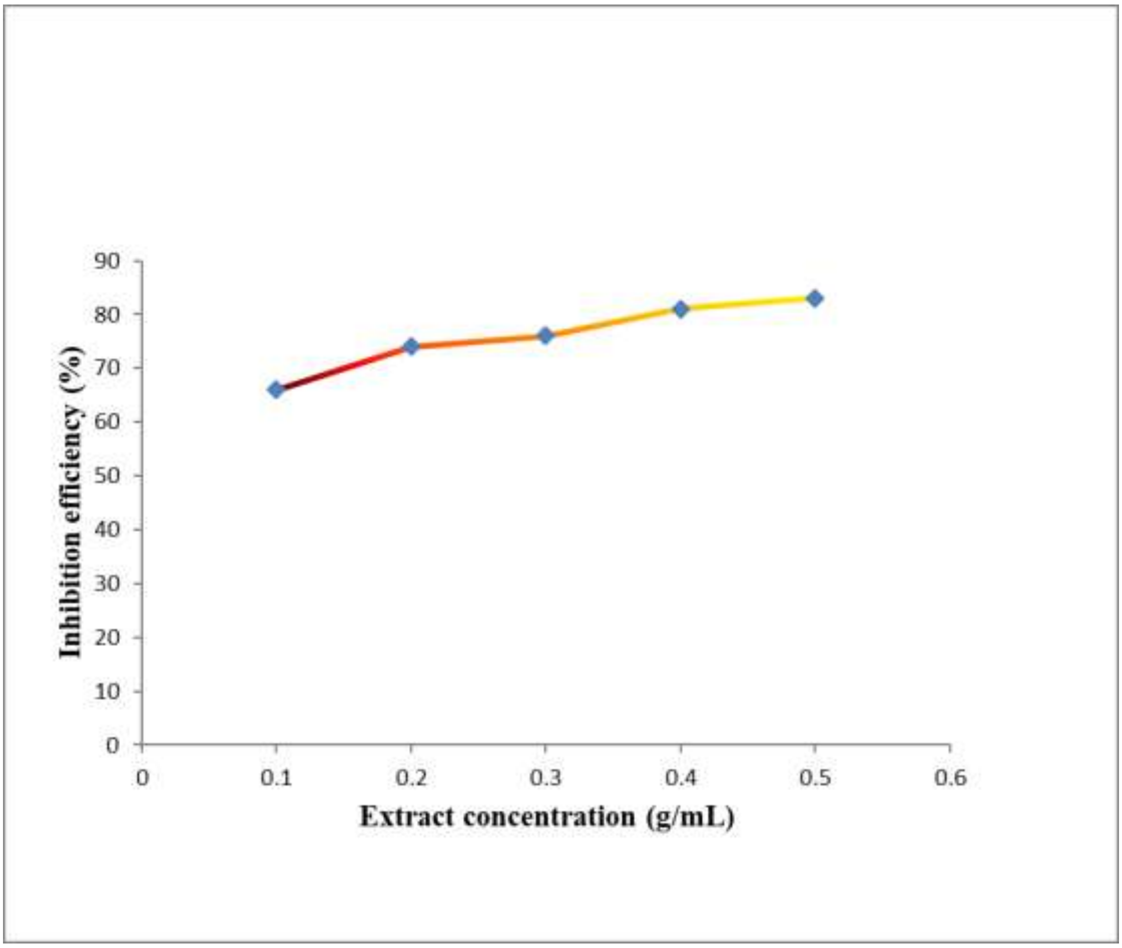

Figure 2: Effect of concentration of *Datura metel* seed extract on the inhibition efficiency of mild steel in 1 M HCl

sites of the metal. The presence of more inhibitors on the metal surface leads to the formation of a thin layer that reduces corrosion process. Similar observations were reported by previous investigators [7, 24].

4.2.3 Effect of temperature on corrosion rate of mild steel

The results of the effect of temperature on the corrosion rate of mild steel in the absence and presence of the extract are shown in Figure 3. It was observed that the rate of corrosion of mild steel increased with increase in temperature, but decreases with the concentration of the extract. This is plausible because corrosion rate increases with increase in the temperature as a result of increase in the average kinetic energy of the reacting molecules. As reported by Fadare *et al*., 2016, the decrease in the corrosion rate in the presence of the extract can be attributed to the inhibitory effect of the extract on mild steel [21].

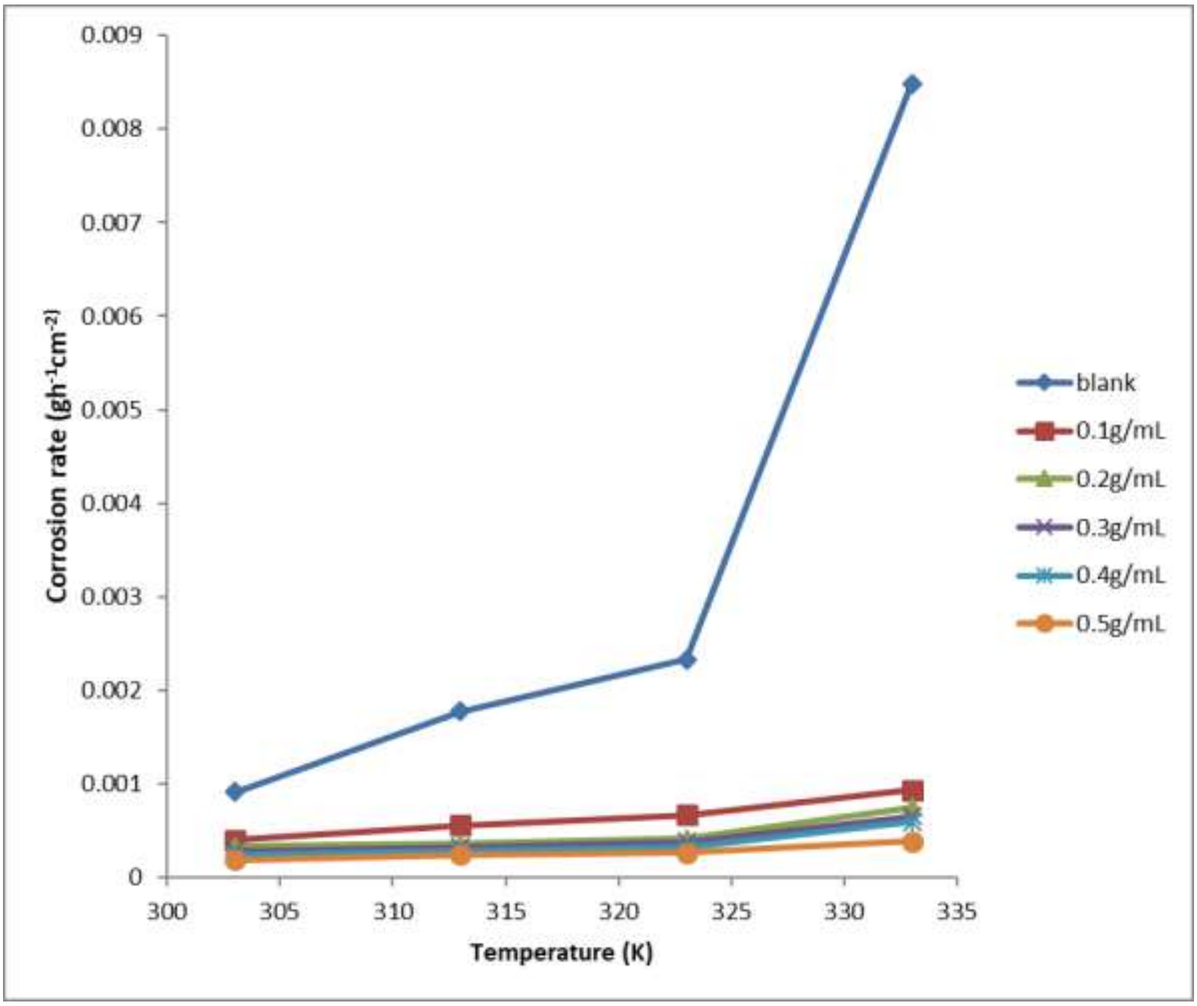

Figure 3: Effect of Temperature on the corrosion rate of mild steel in 1 M HCl

4.2.4 Effect of temperature on inhibition efficiency of mild steel

The inhibition efficiency of ethanol extract of *Datura metel* seed on mild steel in 1 M HCl solution at different temperatures ranging from 303 K to 333 K as shown in Figure 4 clearly revealed that the inhibition efficiency increases with increase in temperature. This indicates that the stability of extract's adsorbed film on the mild steel in acid solution is strongly influenced by temperature [8].

Previous investigators have reported that increase in inhibition efficiency as a result of increase in temperature suggests that the adsorption of the inhibitor on mild steel surface is consistent with the mechanism of chemical adsorption. For a physical adsorption mechanism, inhibition efficiency of an inhibitor is expected to decrease with temperature while a for chemical adsorption mechanism, values of inhibition efficiency increase with temperature [25]. In this study, the inhibition efficiency of ethanol extract of *Datura metel* increases with temperature suggesting that the adsorption of the extract on mild steel surface is consistent with the mechanism of chemical adsorption.

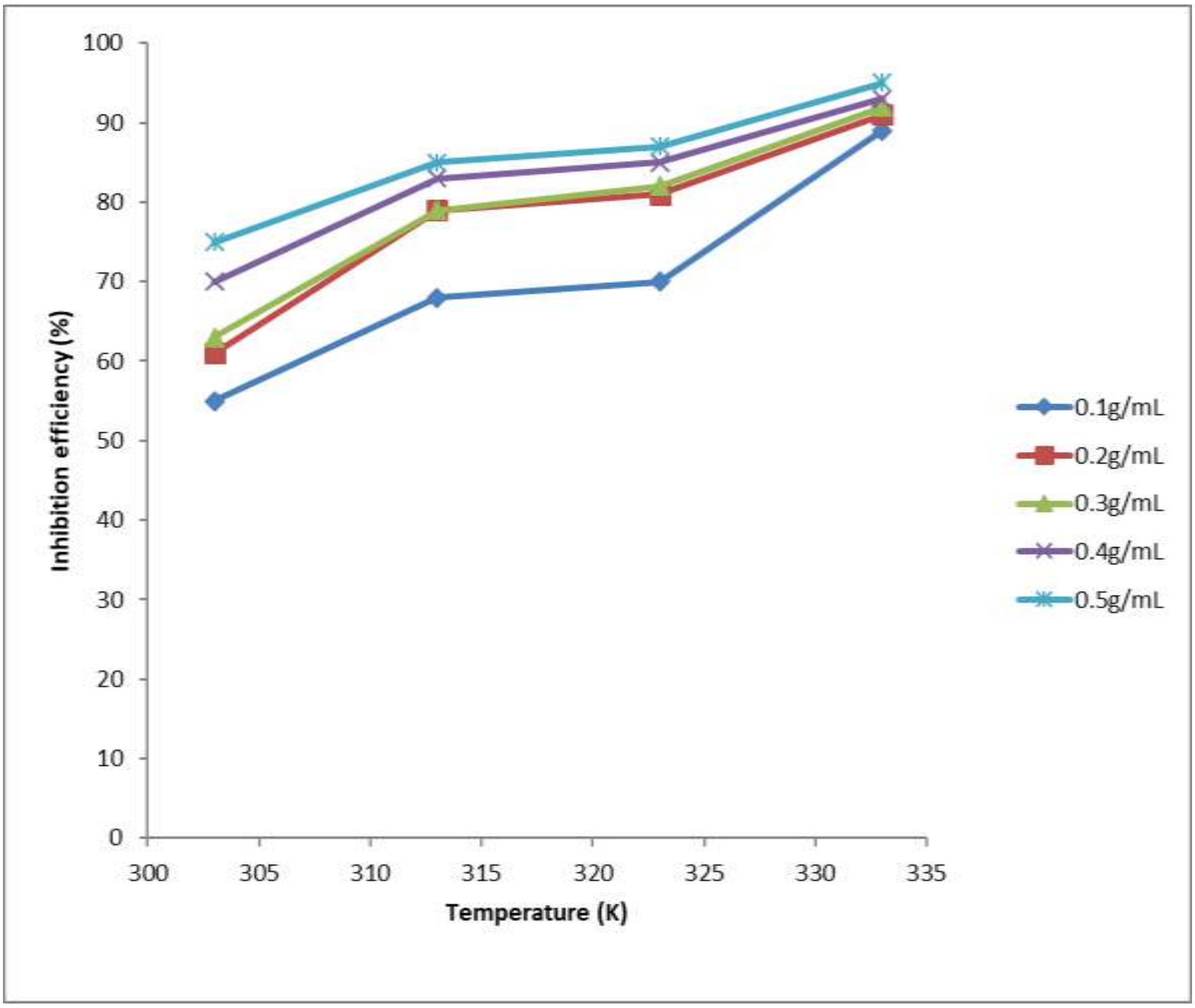

Figure 4: Inhibition efficiency of *Datura metel* seed extract on mild steel in 1 M HCl at various temperatures

3.3 Thermodynamic Studies

Thermodynamic parameters are important to further understand the adsorption process of inhibitor on metal/solution interface [16]. The adsorption of organic compounds can be described by two main types of interactions: physisorption and chemisorption. They are influenced by the nature of the charge of the metal, the chemical structure of the inhibitor, pH, the type of the electrolyte and temperature. Thus, in order to elucidate the inhibitive properties of the inhibitor and the temperature dependence on the corrosion rate, the apparent activation energy (Ea) for the corrosion process in the absence and presence of the inhibitor was evaluated from Arrhenius equation [10].

3.3.1 Determination of activation energy (Ea)

On the basis of Arrhenius equation, the natural logarithm of the corrosion rate (log C_R) is a linear function with 1/T for the acid corrosion of steel. According to equation 4.1, a plot of logarithm of corrosion rate obtained by weight loss measurement versus 1/T gave a straight line graph as shown in Figure 5 with a slope of –Ea/2.303R from which the thermodynamic parameters can be evaluated.

$$\log C_R = \frac{-E_a}{2.303RT} + \log A \qquad 4.1$$

Where C_R is the corrosion rate, Ea is the apparent activation energy, R is the molar gas constant, T is the absolute temperature (in Kelvin) and A is the Arrhenius pre-exponential factor. The results as presented in Table 2 showed that the values of the activation energy are lower in the inhibited solution compared to the un-inhibited solution. It has been reported by researchers that decrease in activation energy values in the presence of inhibitor as observed in this study is actually suggestive of chemical adsorption mechanism while the increase in the Ea in the presence of inhibitor suggests physical adsorption mechanism [26, 27]. In addition, Dehri and Ozean, 2006 reported that for inhibitors whose percentage inhibition efficiency increases with increase in temperature, the activation energy values of the inhibited solution are usually found to be lower than that of the uninhibited solution which is indicative of chemisorption [28]. Conversely, when percentage inhibition efficiency decreases with increase in temperature, and the value of activation energy is found to be higher in the presence of an inhibitor than its value in an uninhibited solution, it is associated with physisorption. In this study, the mechanism of adsorption of ethanol extract of *Datura metel* on mild steel is suggestive of chemical adsorption.

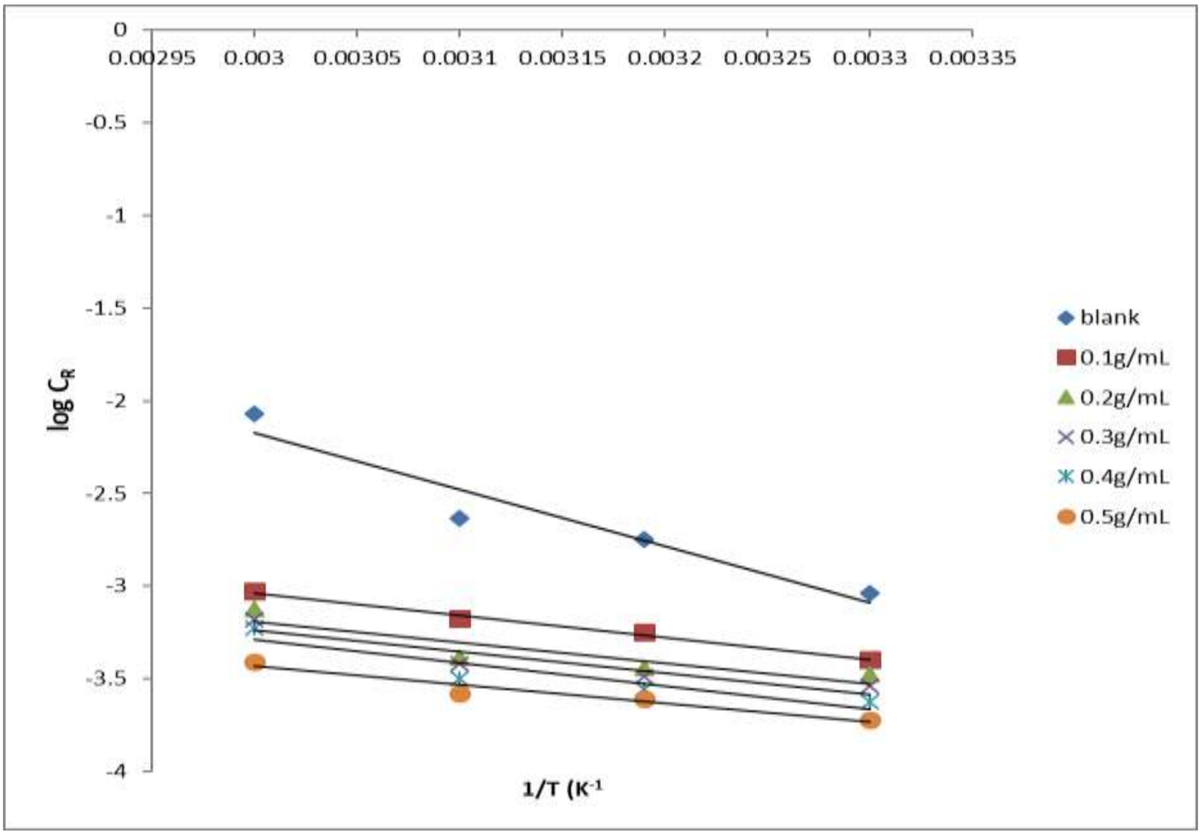

Figure 5Arrhenius plots of log C_R versus 1/T in the absence and presence of different concentrations of *Datura metel* seed extract in 1 M HCl

Table 2: Calculated values of thermodynamic parameters for the corrosion of mild steel in 1M HCl in the absence and present of *Datura metel seed* extract.

Concentration of extract (g/mL)	ΔS (kJ/mol/K)	ΔH (kJ/mol)	Ea (kJ/mol)
Blank	-0.0712	55.2286	58.4458
0.1	-0.1934	20.0118	22.9034
0.2	-0.2007	18.5812	21.2565
0.3	-0.1991	19.3798	22.0991
0.4	-0.1944	21.3140	24.0716
0.5	-0.2127	16.1396	18.9949

3.3.2 Determination of enthalpy (ΔH) and entropy (ΔS)

Other thermodynamic parameters such as change in enthalpy (ΔH) and entropy (ΔS) of the activation process were evaluated from the effect of temperature on the corrosion rate of mild steel in 1 M HCl using the equation below:

$$log\left(\frac{C_R}{T}\right) = log\left(\frac{R}{Nh}\right) + \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303RT} \qquad 4.2$$

Where h is the Planck's constant, N is the Avogadro's number, T is the absolute temperature, R is the universal gas constant, ΔS is the entropy of activation and ΔH is the enthalpy of activation. A plot of log C_R/T versus 1/T (Figure 6) gave a straight line with slope of (-ΔH/2.303RT) and an intercept (log R/Nh) + (ΔS^0/2.303R). Thus, ΔH and ΔS values are evaluated from the slope and intercept, respectively. The results in Table 2 show that the enthalpy of activation are all positive. These positive values of enthalpy reflect the endothermic nature of the steel dissolution process. Also, entropy values are negative indicating that the activation complex in the ratedetermining step represents an association rather than dissociation step. The well oriented inhibitor would have reduced the dissolution of mild steel in HCl solution and thereby inhibiting it from the aggressive medium. These findings were in accordance with Fouda and Ellithy, 2009; Olasehinde *et al*., 2015 [11, 29].

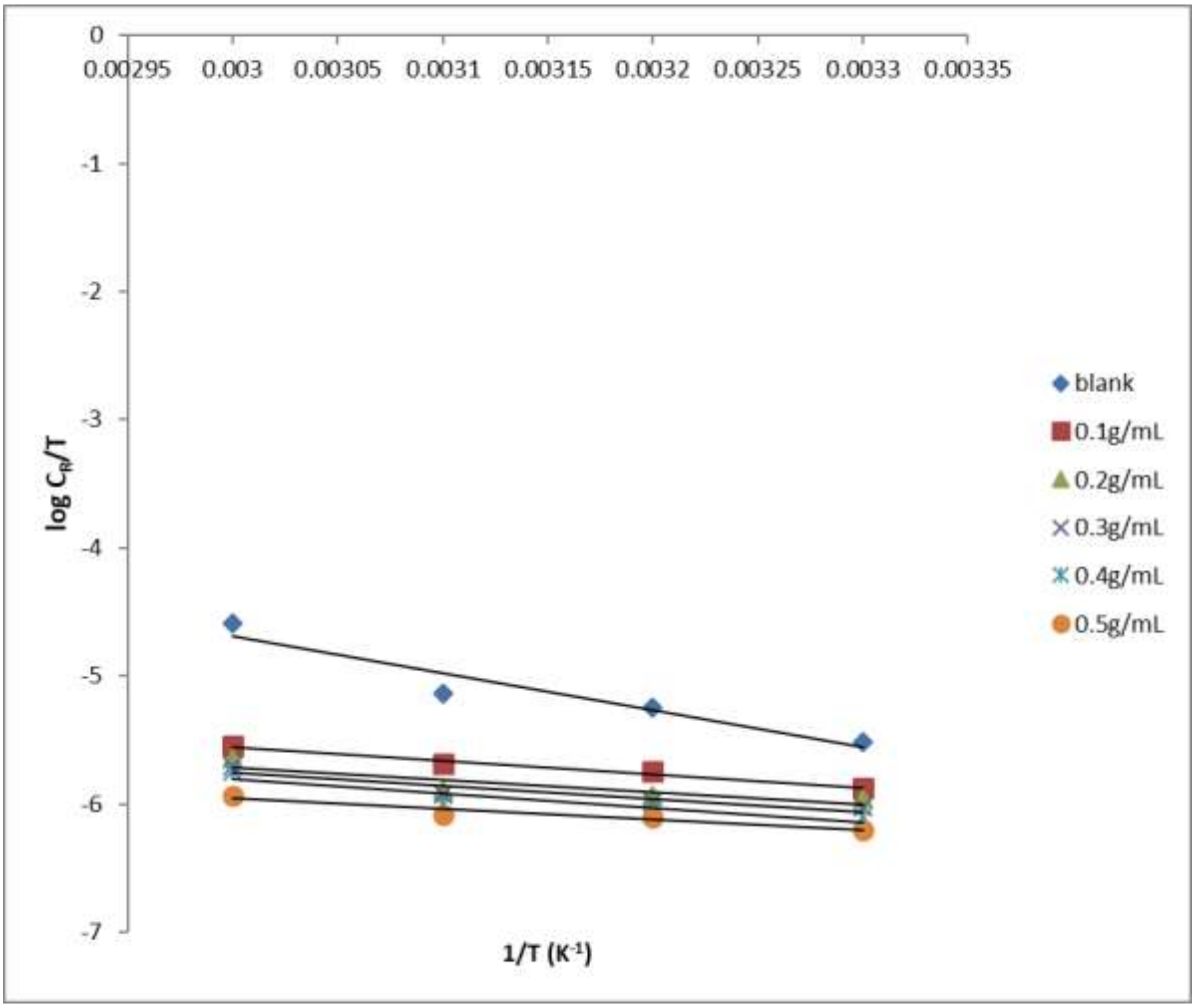

Figure 6 Eyring transition state plot for mild steel in 1 M HCl in the absence (Blank) and presence of ethanol extract of *Datura metel* seed

3.4 Adsorption Isotherm

In adsorption isotherm, surface coverage (ϴ) is very useful as it helps in understanding adsorption characteristics [11]. The degree of surface coverage values for different concentrations of ethanol extract of *Datura metel* seed obtained at different temperatures from weight loss measurement were calculated using the Equation below:

$$\text{Surface coverage } (\Theta) = \frac{\%\,\text{I.E}}{100} \qquad 4.3$$

Adsorption behaviour of extract of *Datura metel* seed can be explained by Temkin, Langmuir and Freundlich adsorption isotherms. Therefore, attempts were made to fit to the various isotherms and it was found that the best fit was obtained with Langmuir isotherm. Langmuir adsorption isotherm describes quantitatively the formation of a monolayer adsorbate on the outer surface of adsorbents, with the assumption that all binding sites have equal affinity for sorbate and the sorption takes place at specific homogeneous sites within the adsorbents [30]. It can be expressed according to the equation below:

$$\frac{C}{\theta} = \frac{1}{k_{ads}} + C \qquad 4.4$$

Where C is the concentration of the inhibitor, K_{ads} is the adsorption equilibrium constant and ϴ is the degree of surface coverage of the inhibitor.

As shown in Figure 7, the plot of C versus C/θ gives a linear correlation and the value of C/ϴ increases as the concentration of the extract increases. The strong correlation ($R^2 \approx 0.99$) for all the extract concentrations and at different temperature suggests that the adsorption of inhibitor on the mild steel surface obeyed this isotherm. This indicates that the adsorbing *Datura metel* seed extract occupies typical adsorption site at the metal/solution surface. In this study, the Langmuir isotherm characterizes chemisorption of the adsorbed species and postulates monolayer adsorption of the adsorbate onto the adsorbent which is expected to have a slope of unity [8]. The slope of unity obtained in this study is an indication that the adsorption of the components is approximated by Langmuir adsorption isotherm and that the monolayer of the inhibitor species must have been attached to mild steel surface without lateral interaction between adsorbed species.

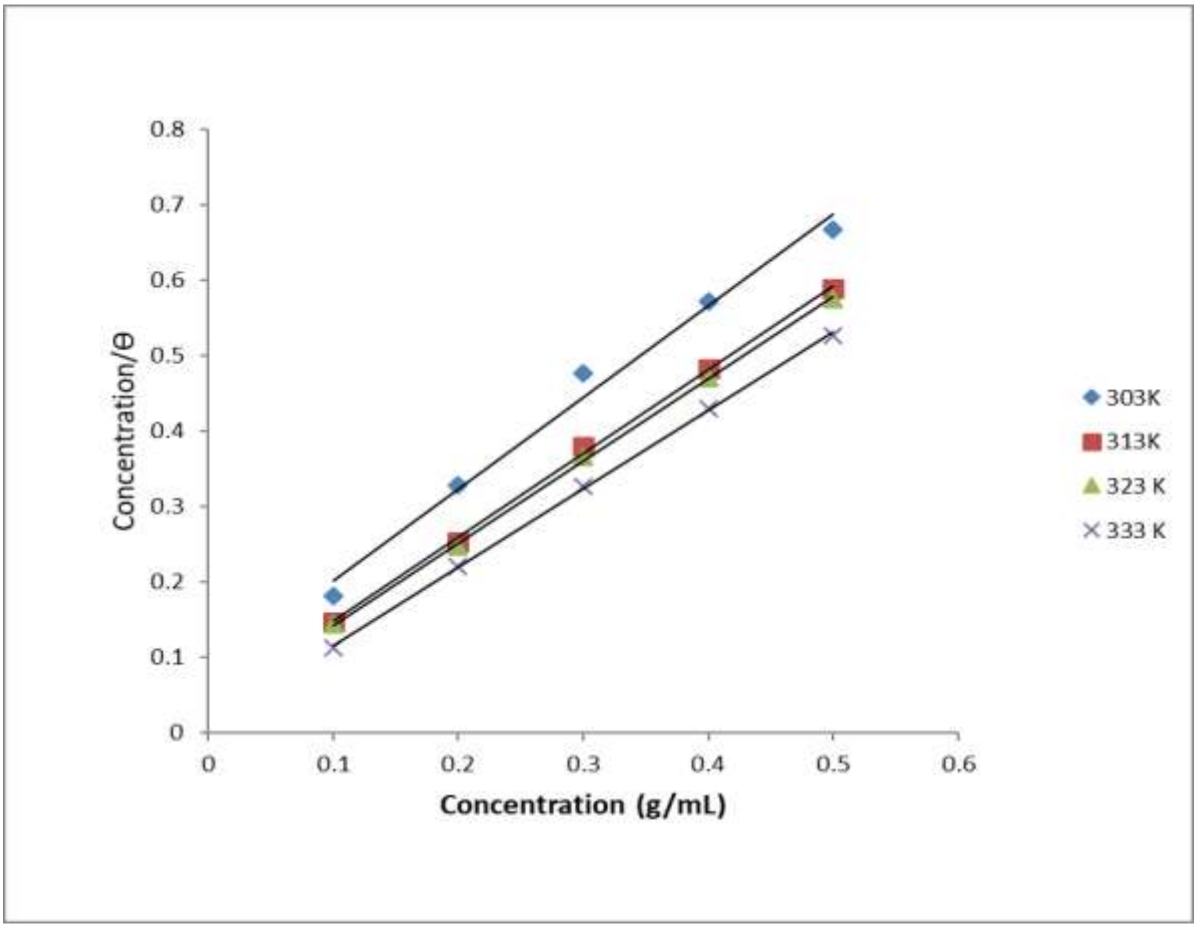

Figure 7Langmuir adsorption isotherm plot for mild steel corrosion in 1 M HCl for ethanol extract of *Datura metel* seed at different temperatures

Table 3 Calculated parameters from Langmuir adsorption isotherm plot for *Datura metel* seed extract

Temperature (K)	Slope	R^2 values	K_{ads}	(ΔG) (kJ/mol)
303	1.213	0.987	12.500	-16.4835
313	1.110	0.998	27.778	-19.1079
323	1.087	0.999	29.412	-19.8687
333	1.038	0.999	90.909	-23.6094

3.4.1 Free energy determination

The free energies of adsorption, ΔG_{ads} were calculated from the equilibrium constant of adsorption using the equation;

$$K_{ads} = \frac{1}{55.5} \exp\left(\frac{-\Delta G_{ads}}{RT}\right) \qquad 4.5$$

This equation can also be expressed as follows:

$$\Delta G_{ads} = -2.303RT \log (55.5K_{ads}) \qquad 4.6$$

Where R is the universal constant, T represent absolute temperature, 55.5 indicates molar concentration of water and K_{ads} is the adsorption constant. As shown in Table 3. The values of K_{ads} were found to increase with increase in temperature showing that the interaction between the adsorbed molecules and the metal surface becomes strengthened and that the inhibition molecules possess strong interaction with the metal surface, which results in an increase in the inhibition efficiency as temperature increases.

3.5 Fourier Transform Infrared Spectroscopy (FT-IR)

The chemical constituents that contribute to the corrosion inhibition have specific functional groups [31]. The extract and the adsorption were analysed with FT-IR to identify the functional groups and the results are given in Figures 8 and 9, respectively. According to Figure 8, absorption band was observed at 3354.32 cm^{-1}corresponding to primary amine N–H stretch, 2928.11 $cm^{-1}$and 2854.74 cm^{-1}confirming the characteristic doublet bands of aldehyde C–H stretch, 2353.16 cm^{-1}corresponding to cyano group C≡N, 2150.20 cm^{-1}confirming the presence of alkyne C≡C, 1745.64 cm^{-1}corresponding to aldedyde carbonyl group C=O, while 1653.05 $cm^{-1}$and 1024.24 cm^{-1}correspond to amide carbonyl group C=O and ether C–O–C respectively.

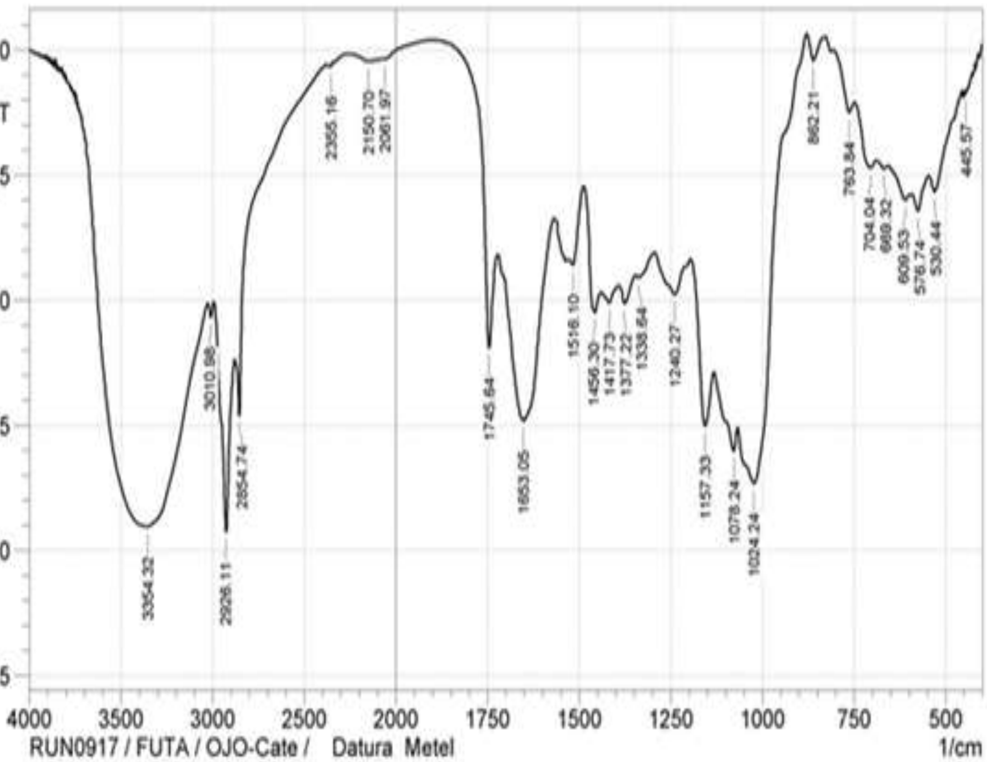

Figure 8FT-IR spectrum of ethanol extract of *Datura metel* seed

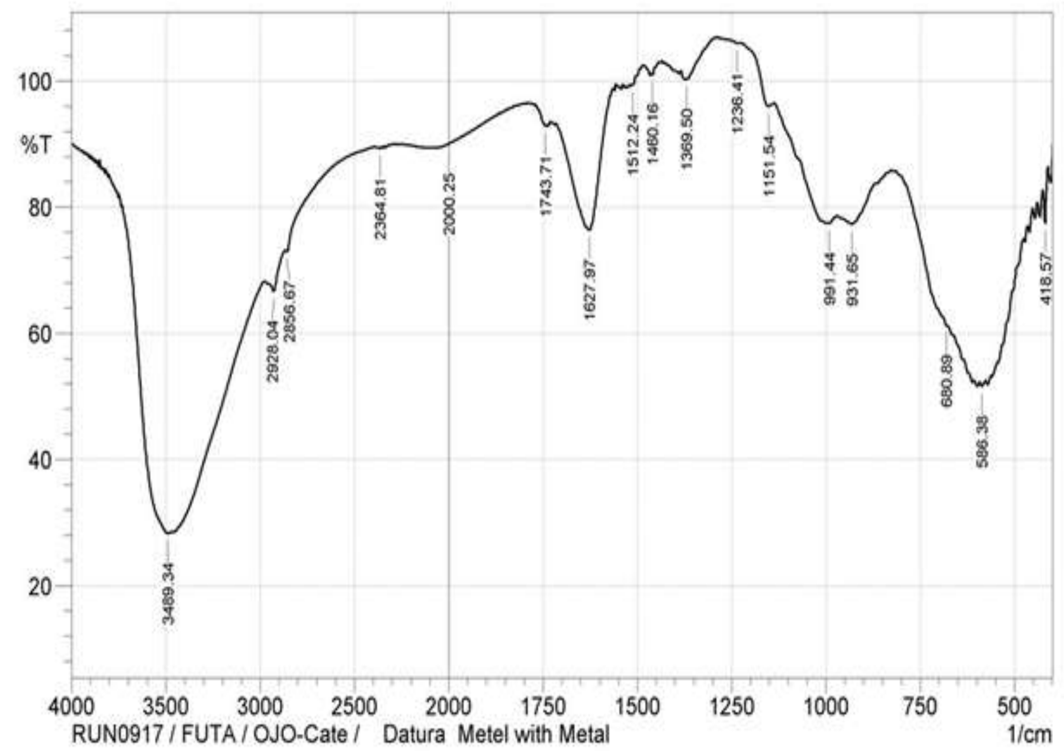

Figure 9: FT-IR spectrum of dried adsorption product of ethanol extract of *Datura metel* seed on mild steel.

Figure 4.9 reveals the absorption bands observed at 3489.34 cm^{-1}, 2928.04 cm^{-1}, 2856.67 cm^{-1}, 2364.81 cm^{-1}, 2000.25 cm^{-1}, 1743.71 cm^{-1}, 1627.97 $cm^{-1}$and 1151.54 cm^{-1}. On comparing the IR spectra of inhibitor only (Figure 8) with that of the adsorption product (Figure 9), there were forward shift from 3489.34 $cm^{-1}$to 3354.32 cm^{-1}corresponding to amino group, backward shift from 2928.11 cm^{-1} and 2854.74 cm^{-1} to 2928.04 $cm^{-1}$and 2856.67 cm^{-1}corresponding to aldehyde, forward shift from 2353.16 $cm^{-1}$to 2364.81 cm^{-1}revealing the presence of cyano group, backward shift from 2150.70 $cm^{-1}$to 2061.97 cm^{-1}corresponding to alkyl group, backward shift from 1745.64 $cm^{-1}$to 1743.71 cm^{-1}corresponding to aldehyde carbonyl group, backward shift from 1653.05 $cm^{-1}$to 1627.95 cm^{-1}corresponding to amide carbonyl group, while forward shift from 1024.24 $cm^{-1}$to 1151.54 cm^{-1}corresponds to ether group.

The functional groups such as amide, aldehyde, cyano, aldehyde and ether which were observed indicating that the plant extract contains the mixture of compounds that can facilitate the transfer of electron from the inhibitor molecule to the vacant d-orbital of the metal. Organic substances containing polar functions with oxygen, nitrogen and/or sulphur atoms in a conjugated system have been reported to exhibit good inhibiting properties [32, 33]. The shifting in the frequencies may imply that the active phytochemical constituents present in the inhibitor molecules bind to the metal surface to form a protective metal inhibitor complex which reduce the further dissolution of metal in the aggressive media [34].

3.6 Atomic Absorption Spectroscopy Analysis

The plot of the dissolution of iron (II) ion against various concentration of *Datura metel* seed extract is shown in Figure 10 It was observed that there was a steady reduction in the concentrations of Fe^{2+}in solution as the concentration of the extract increased. This reduction in the concentration of $Fe^{2+}$is due to the adsorption of the extract on the surface of the iron filling in the acidic media which mitigated the rapid oxidation of Fe to Fe^{2+}. Hence, the reduction observed in the Fe^{2+}concentration in inhibited solution shows that the extract has been able to retard the corrosion rate of the iron in the acid solution. Figure 10 shows the amount of Fe in the acid in the presence and absence of inhibitor. The percentage of Fe^{2+} in the blank is the highest while the percentage of Fe^{2+} decreases as the concentration of inhibitor increases. This observation is in consonance with the previous investigators. [21, 35].

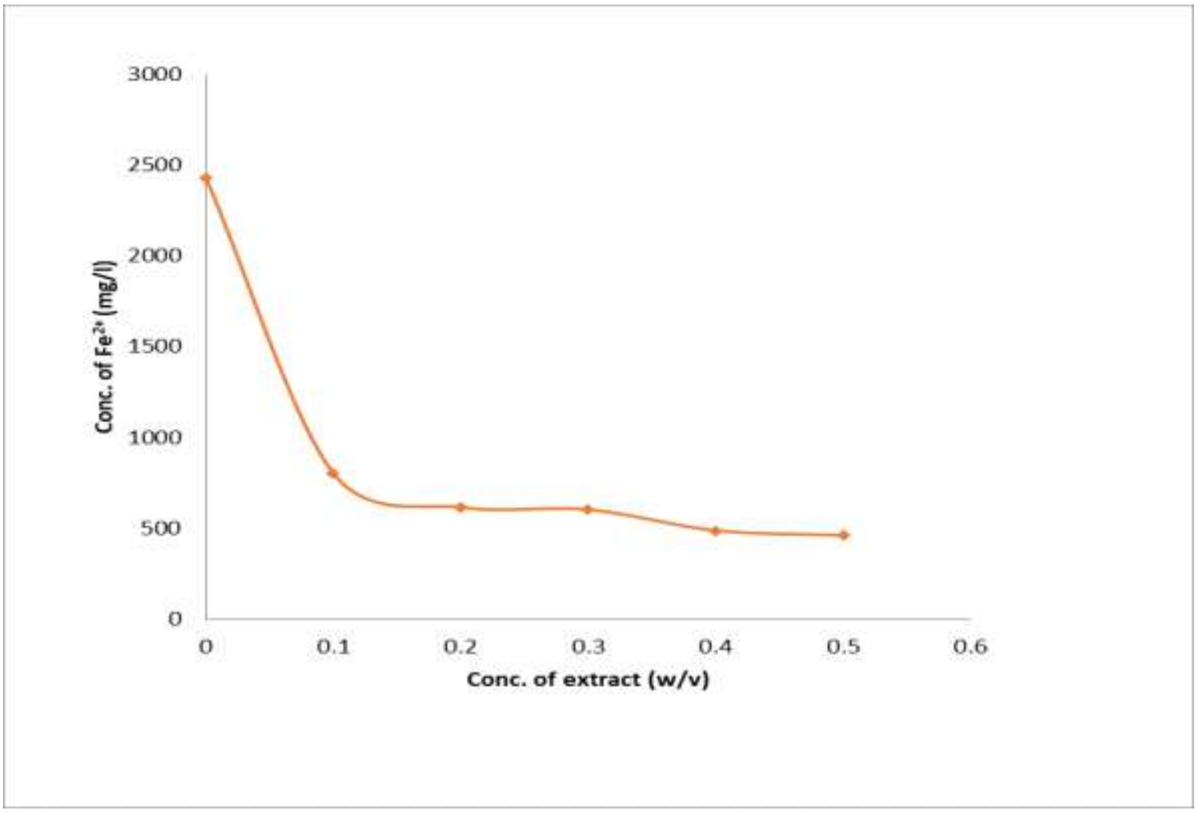

Figure 10: AAS analysis of the effect of *Datura metel* seed extract concentration on the iron (II) dissolution into acidic solution.

3.7 Surface Morphological Studies

Surface analysis of mild steel using Scanning Electron Microscope/Energy Dispersive X-ray (SEM-EDS) provided information on the level of attack as well as the strength of inhibition of the extract on mild steel surface. Analysis of elemental composition on the surface was also examined by SEM-EDS. The results are presented in Figures 11, 12, 13, 14, 15 and 16 respectively. Figure 11 and 14 shows the micrograph and the EDS result of the mild steel strip after smoothening the surface with emery paper, before its immersion in 1 M HCl acid. Figure 11 shows a clear and smooth surface while Figure 14 shows the weight percent of Fe as 83.69%. Figure 12 and 15 shows the micrograph and the EDS result of the mild steel strip after its immersion in 1 M HCl for 4 hours. On comparing the micrograph and EDS result of the mild steel before immersion into acid (Figure 11 and Figure 14) with that after its immersion into acid (Figure 12 and Figure 15), the effect of the acid is clearly observed on the mild steel because

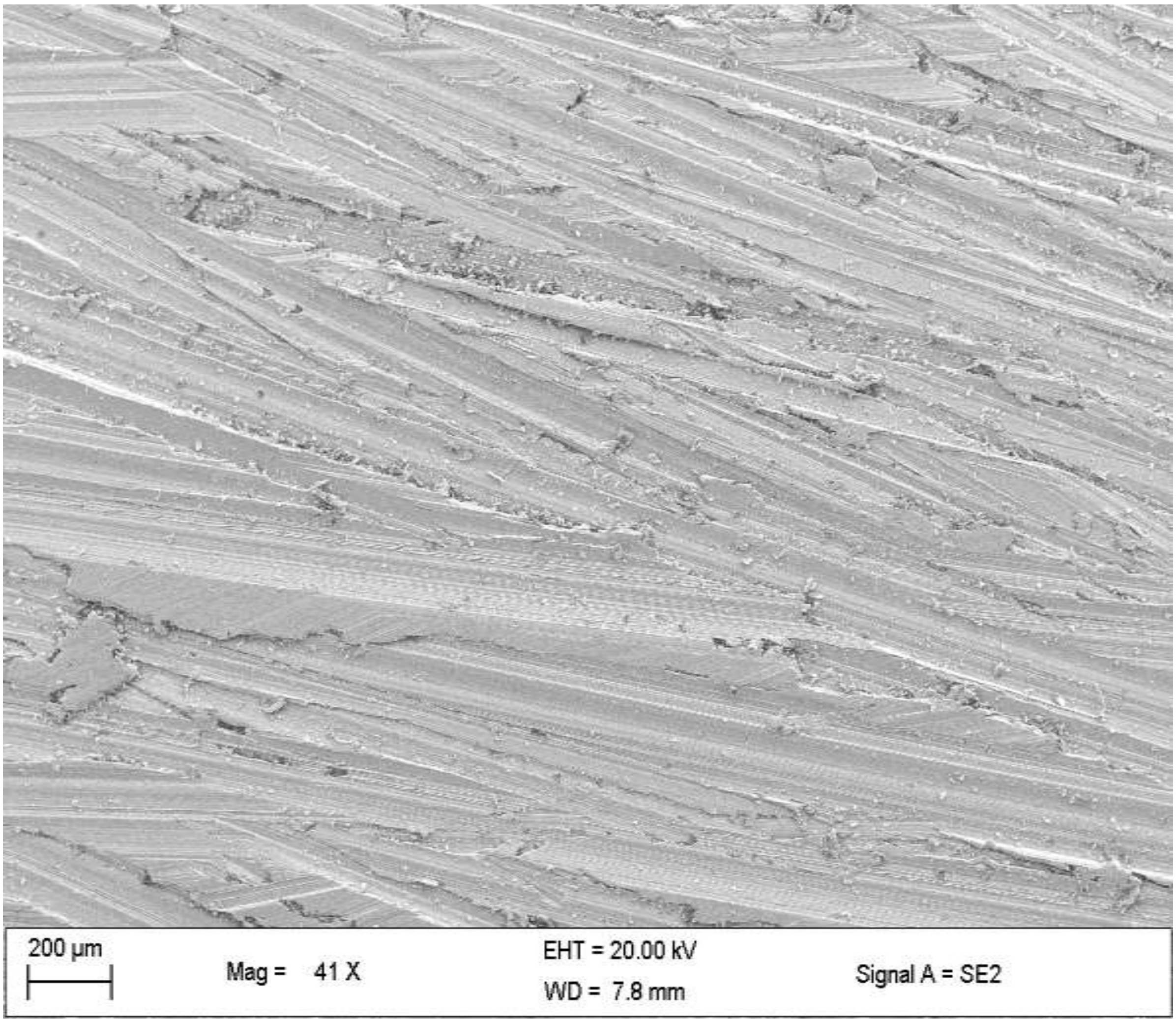

Figure 11: Surface morphology of mild steel sample prior to corrosion study

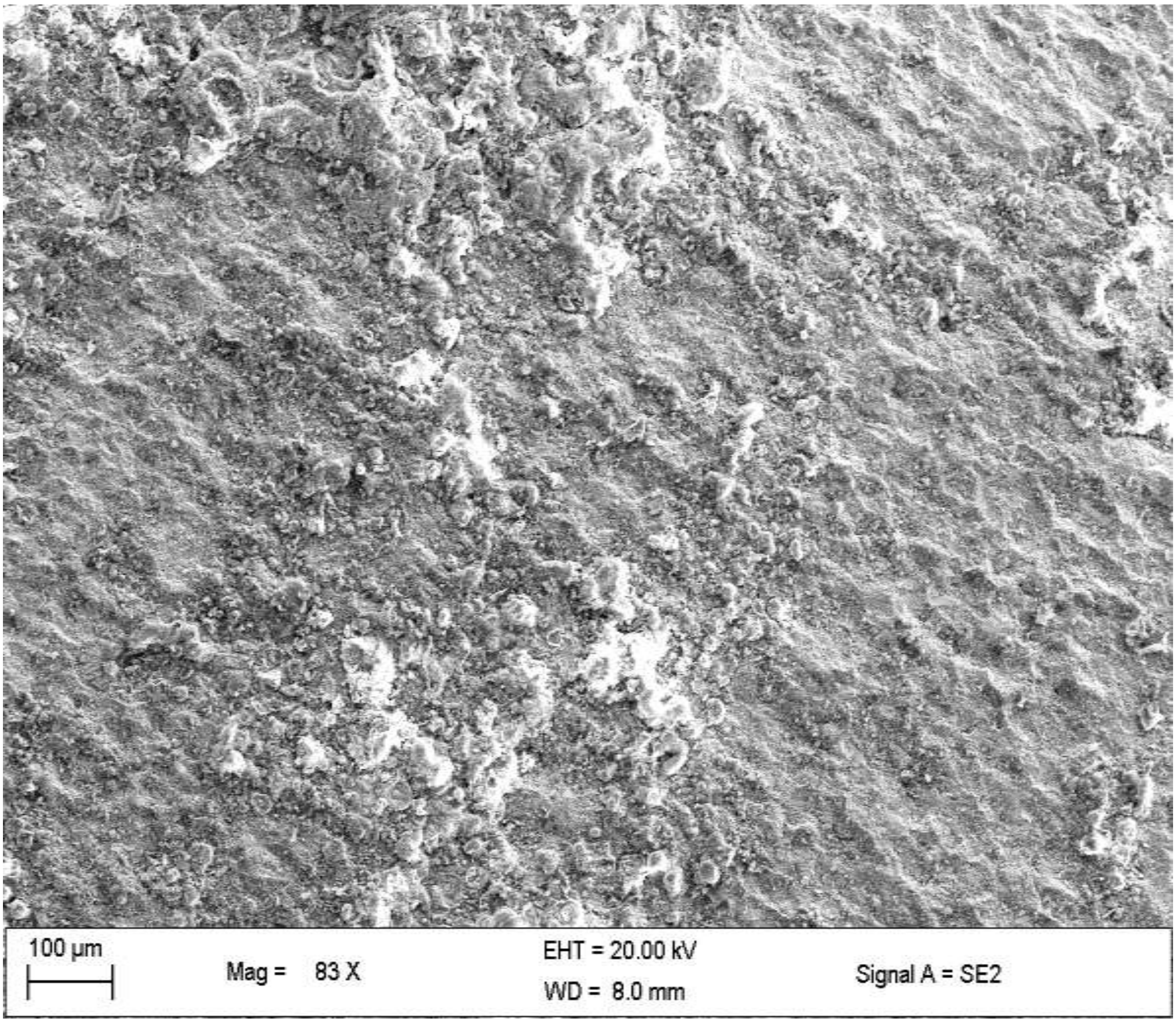

Figure 12: Surface morphology of mild steel sample after immersion in 1 M HCl solution without inhibitor

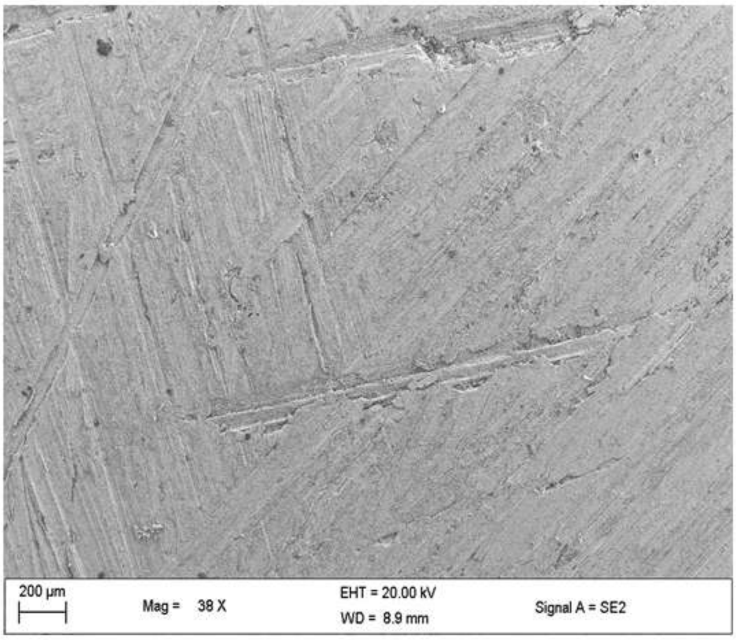

Figure 13: Surface morphology of mild steel sample after immersion in 1 M HCl solution with the addition of *Datura metel* seed inhibitor

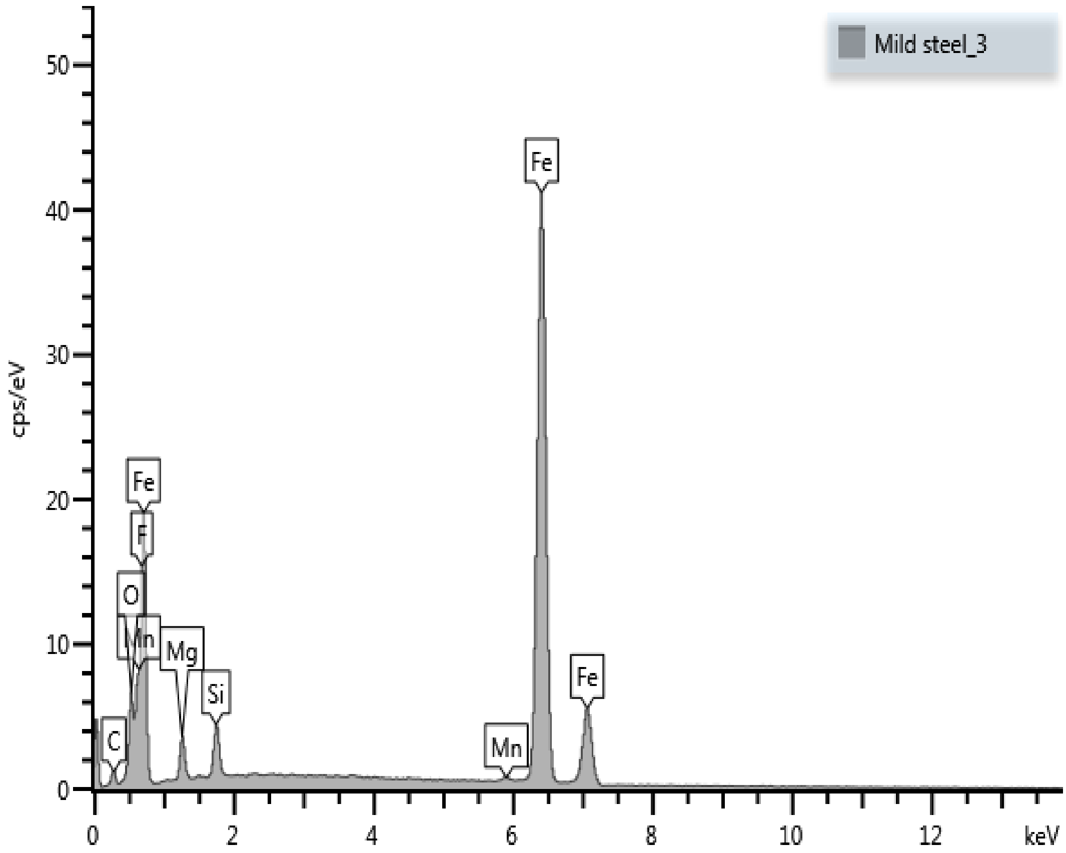

Figure 14: EDS spectra of mild steel sample prior to corrosion study.

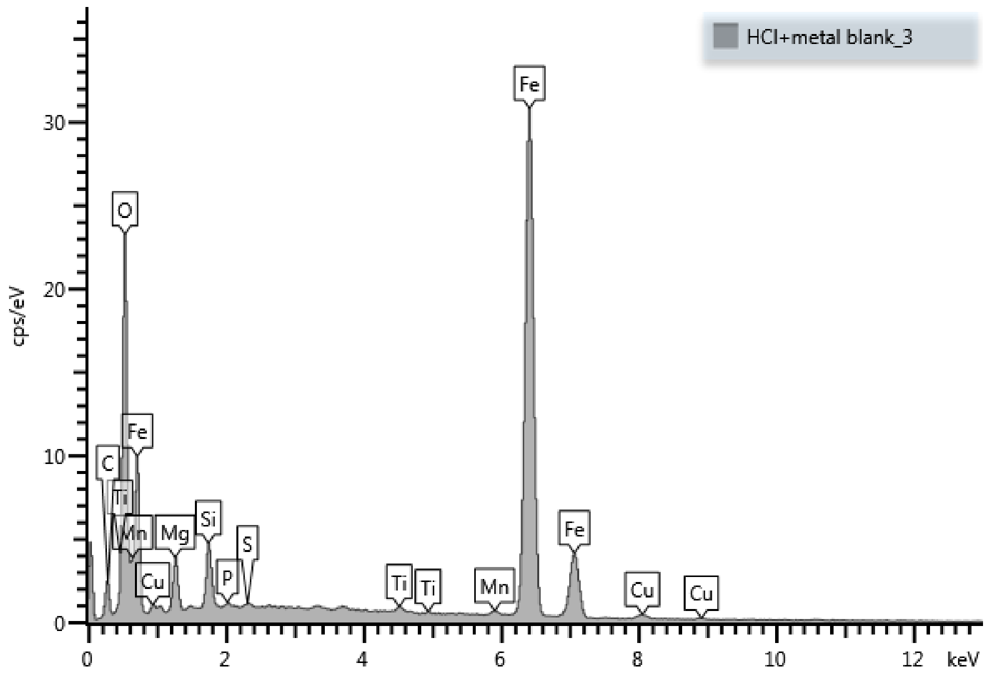

Figure 15: EDS spectra of mild steel sample after immersion in 1 M HCl solution without inhibitor.

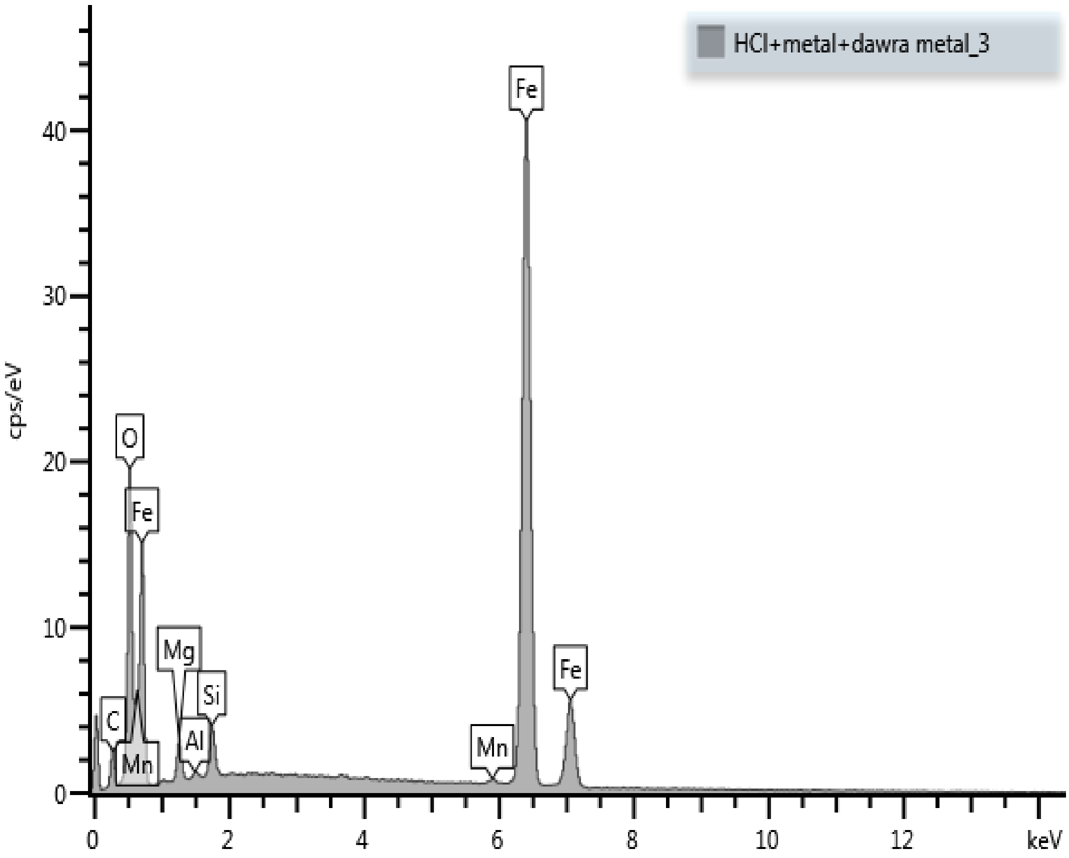

Figure 16: EDS spectra of mild steel sample after immersion in 1 M HCl solution with the addition of *Datura metel* seed inhibitor.

the surface of the metal has been cracked suggesting corrosion that has taken place. Also, there is a change in the weight percent of Fe from 83.69% to 59.55% showing that corrosion of metal has taken place in the acid solution. Figure 13 and 16 shows the micrograph and the EDS result of the mild steel after immersion in 1 M HCl acid solution containing *Datura metel* seed extract. The extract mitigated the corrosion of the mild steel to a large extent as revealed by the micrograph and the EDS result. The mild steel surface in the free acid solution was cracked due to the acid attack on the surface (Figure 12). However, in the presence of 0.5% (g/mL) *Datura metel* seed extract, the roughness was drastically reduced and the weight percent of the Fe in the mild steel was not as reduced as that in the blank. The weight percent of Fe reduces from 83.69% to 72.73%. This EDS result shows that the inhibitor has retarded the dissolution of the mild steel in the acid solution. The inhibition efficiency calculated from this concentration was 83%. From this micrograph, it is evident that the average roughness of the surface of the metal was drastically reduced compared to the micrograph in (Figure 12) the blank. This is in line with various studies on plant extracts [14, 21, 36].

3.8 Electrochemical Study

The potentiodynamic polarization curves for the mild steel in 1M HCl solution in the absence and presence of various concentrations of *Datura metel* seed extract is shown in Figure 17.

The values of the corrosion current density (I_{corr}), the corrosion potential (E_{corr}), cathodic tafel slope (βc), anodic tafel slope (βa) and the percentage of inhibition efficiency (I.E %) obtained from the polarization curves for the extract are shown in Table 4.

The percentage inhibition efficiency was obtained by using the equation 4.7

$$I.E\% = \frac{Icorr - Icorr(inh)}{Icorr} \times 100 \qquad 4.7$$

Where Icorr and I_{corr}(inh) are the corrosion current density values without and with inhibitor respectively. From Table 4, it is evident that the highest inhibition efficiency obtained for the ethanolic extract of *Datura metel* seed is 91.39% suggesting that these plant extracts could serve as effective green corrosion inhibitors. Also, it is noticed that the addition of the extract to acid solution decreased the corrosion current density (I_{corr}).

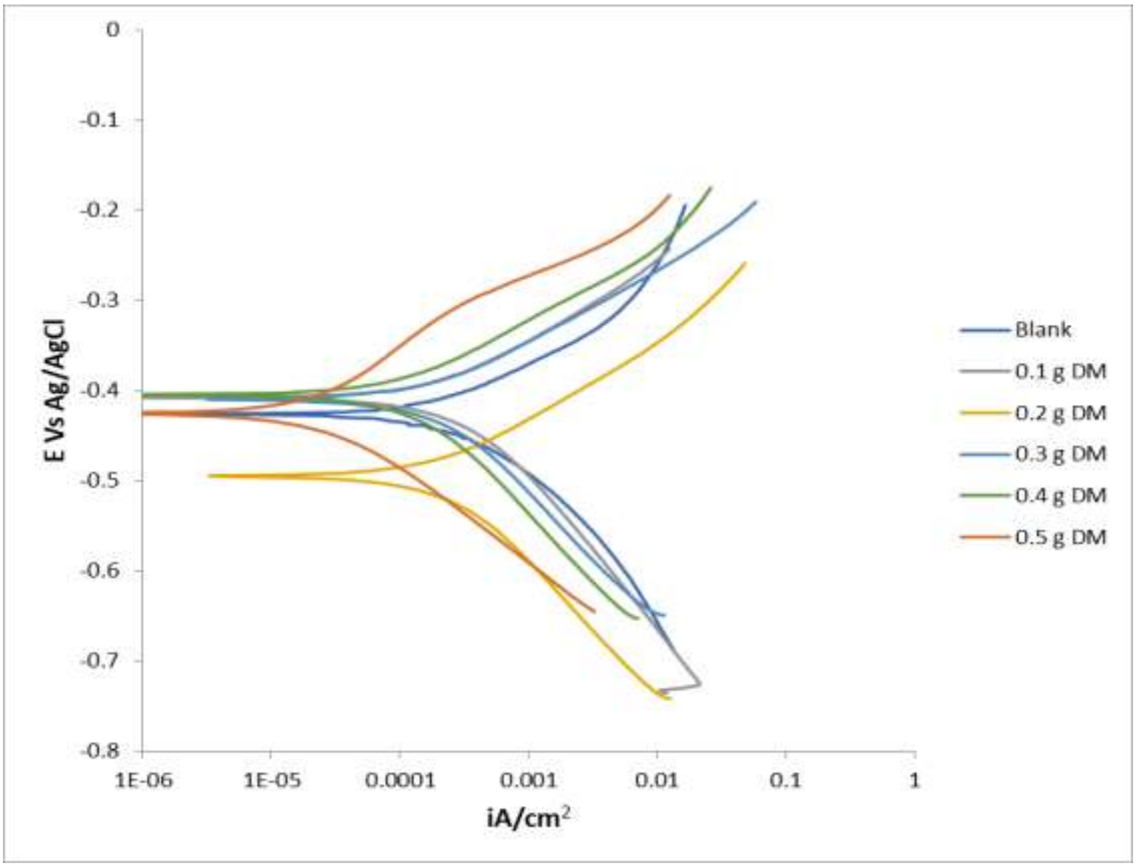

Figure 17: Potentiodynamic polarization curves for mild steel in 1 M HCl without and with various concentration of *Datura metel* seed extract.

Table 4: Potentiodynamic polarization parameters for mild steel in 1 M HCl in absence and presence of *Datura metel* seed extract.

Extract conc. (g/mL)	E_{corr} (mv)	I_{corr} ($\mu A/cm^2$)	βa (mv)	βc (mv)	Corrosion rate Cr (mmpy)	I.E (%)
Blank	-426.515	317.403	97.663	136.923	3.6830	-
0.1	-408.140	282.154	108.410	153.504	3.2740	11.11
0.2	-495.238	243.069	97.133	150.607	2.8205	23.42
0.3	-409.605	201.925	87.773	148.950	2.3421	36.38
0.4	-403.662	127.475	88.576	149.185	1.4792	59.84
0.5	-425.628	27.324	123.648	102.061	0.3284	91.39

The decreasing I_{corr} indicates the success of suppression of corrosion from the metal dissolution which is attributed to the increase in adsorption of inhibitor molecules on the metal surface inhibiting the charge transfer from the anodic and cathodic reaction [31]. It has been reported that (i) if the displacement in E_{corr} > 85 mV with respect to E_{corr} of the blank (uninhibited solution), the inhibitor can act as a cathodic or anodic type and (ii) if the displacement in E_{corr} is< 85 mV, the inhibitor can be considered as a mixed type [37]. In the present study, the shift in E_{corr} values is in the range -403.662mV to -426.515 mV which is < 85 mV. Since the displacement of the corrosion potential is not up to 85mV (Table 4) after the addition of the extracts, it suggests that the inhibitor acts as a mixed-type inhibitor with predominant cathodic effectiveness. Also, it is evident that the addition of ethanol extract of the seed in 1 M HCl alter both anodic and cathodic tafel slope (βa and βc) values indicating that the presence of the extract inhibits both cathodic and anodic reactions and the extract can thus be classified as mixed corrosion inhibitor. This report is in agreement with the previous investigator [38].

IV. Conclusion

The study has shown the ethanol extract of *Datura metel* seed as effective corrosion inhibitor for mild steel in 1 M HCl solution. Phytochemical screening revealed the presence of many important phytoconstituents such as; saponins, tannins, alkaloids, phenols, e.t.c. which are reported as anticorrosive agents. This was supported by functional groups confirmed by FT-IR analysis. The percentage inhibition efficiency increases with increase in the extract concentration. This can be attributed to the increase in the number of constituent molecules adsorbed on the metal surface at high concentration so that the active sites of the metal are protected by the inhibitor molecules. However, the inhibition efficiency of the extract increases with rise in temperature and its increase leads to decreased activation energy of the corrosion process, which suggests a chemical adsorption mechanism. Thermodynamic studies further corroborate that the mechanism of adsorption is chemisorptions. The adsorption of the inhibitor on mild steel surface was best fitted to Langmuir adsorption isotherm and the free energy indicates that the process was spontaneous. AAS analysis showed a decrease in the dissolution of the mild steel with increase in extract concentration. The SEM images of the mild steel samples showed that the metal was protected in the presence of the extract. Potentiodynamic polarization measurements demonstrated that the extract acts as a mixed type inhibitor.

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