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The Effects of Inhibition on Corrosion of Mild Steel in H_2SO_4 Using Ethanol Extract of Vernonia Amygdalina

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ABSTRACT

In this paper, adsorption and thermodynamic study of the inhibition of corrosion of mild steel in 1M H_2SO_4 medium using ethanol extract of vernonia amygdalina was carried out using weight loss and IR methods of monitoring corrosion. The results reveal that ethanol extract of vernonia amygdalina inhibited corrosion of mild steel. The inhibition efficiency ranged from 83.91% to 89.11%, 81.09 % to 85.00%, 81.08% to 84.90% and 77.45% to 79.66% at 303, 313, 323 and 333K respectively. The inhibition efficiency of the extract increased as the concentration of the extract increases but decreased as temperature and time of immersion increases. The adsorption of the inhibitor on the surface of mild steel was found to be exothermic, spontaneous and consistent with the mechanism of physical adsorption as the value for heat of adsorption ranged from -35.22 to -42.90J/mol. The adsorption of the inhibitor followed the Langmuir isotherm. IR spectra of the corrosion product (without inhibitor), the extract and the corrosion product (with the inhibitor) confirmed that ethanol extract of vernonia amygdalina is an adsorption inhibitor.

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I. Introduction

In the world today, mild steel is used in different Engineering applications for the production of some automobile components, structural shapes and sheets that are used in pipelines, buildings, plants, bridges and tin cans.[1] The study of corrosion of mild steel and iron is a matter of tremendous theoretical and practical concern and as such has received a considerable amount of interest. Acid solutions, widely used in industrial acid cleaning, acid descaling, acid pickling, and oil well acidizing, require the use of corrosion inhibitors in order to restrain their corrosion attack on metallic materials. Mild steel is commonly used for industrial structures due to its strength, ductility, weldability and because of it's amenable to heat treatment for varying mechanical properties

The corrosion of metals remains a worldwide scientific problem as it affects the metallurgical, chemical and oil industries. The increasing interest in the manufacture of sulphuric acid has created the need for obtaining information on the corrosion resistance of mild steel to sulphuric acid attack.[2] Mild steel as a constructional material is often exposed to acidic environment to a great extent during service. This exposure can be under condition of varying temperature, flow rate, pH and other factors; all of which can alter the rate of corrosion. The relative acidity of the solution is the most important factor to be considered; at low pH, the evolution of hydrogen tends to eliminate the possibility of protective film formation so that the steel continues to corrode but in alkaline solutions, the formation of protective film greatly reduces corrosion rate. [3]

Corrosion control of metals is of technical, economic, environmental, and aesthetical importance. The use of inhibitors is one of the best options of protecting metals and alloys against corrosion. The environmental toxicity of organic corrosion inhibitors has prompted the search for green corrosion inhibitors as they are biodegradable, do not contain heavy metals or other toxic compounds. As in addition to being environmentally friendly and ecologically acceptable, plant products are inexpensive, readily available and renewable. Investigations of corrosion inhibiting abilities of tannins, alkaloids, organic, amino acids, and organic dyes of plant origin are of interest. In recent years, sol-gel coatings doped with inhibitors show real promise. Although substantial research has been devoted to corrosion inhibition by plant extracts, reports on the detailed mechanisms of the adsorption process and identification of the active ingredient are still scarce. Development of computational modeling backed by wet experimental results would help to fill this void and help understand the mechanism of inhibitor action, their adsorption patterns, the inhibitor metal surface interface and aid the development of designer inhibitors with an understanding of the time required for the release of self-healing inhibitors.

In other to reduce the corrosion of metals, several techniques have been applied. The use of inhibitors during acid pickling procedure is one of the most practical methods for protection against corrosion in acidic environment. [4] Most of the efficient and effective organic inhibitors are those compounds containing hetero-atoms in their aromatic or long carbon chain. [5] To be effective, an inhibitor must displace water from the metal surface, interact with anodic or cathodic reaction sites to retard the oxidation and reduction corrosion reaction, and prevent transportation of water and corrosion active species on the surface. [4,6]

The environmental consequences of the corrosion are enormous and its inhibition has deeply investigated. It has been found that one of the best methods of protecting metals against corrosion involves the use of the inhibitors which are substances that slow down the rate of corrosion. [7,8,9,10] An inhibitor is added in small amount in other to slow down the rate of corrosion through the mechanism of adsorption. Over the years, several inhibitors have been synthesized or chosen from existing compounds and it has been found that the best inhibitors are those that have center for π electron donation (usually enhanced by the presence of hetero atoms in aromatic compounds) while others may be gotten from extracts of naturally occurring compounds. [7]

Several researches have been done on the use of extract of plant as inhibitor for metals against corrosion in different aggressive media [11]. However, not much has been reported on the use of the extract of Vernonia Amygdalina plant as inhibitor against corrosion of mild steel in H_2SO_4. This paper is aimed at investigating the effect and efficiency of Vernonia Amygdalina in inhibition of mild steel in acidic environment. To achieve this aim, the following objectives have to be met: Determining the potential of ethanol extract of *Vernonia Amygdalina* leaves as corrosion inhibitor of mild steel in tetraoxosulphate (VI) acid, determining the effect of temperature (303 – 333K) on the corrosion of mild steel, determining the effect of different concentrations of ethanol extract of *Vernonia Amygdalina* on the corrosion of mild steel in 1M tetraoxosulphate (VI) acid at different temperatures (303 – 333K), Calculating the kinetic, thermodynamic and adsorption parameters of the inhibitor in order to correlate theoretical/calculated inhibition efficiencies with experimental results, and Propose the mechanism of the corrosion of mild steel in H_2SO

II. Materials And Methods

1.1. Materials

Mild steel sheets of composition (wt %) Mn (0.6), P(0.36), C(0.15) and Si(0.03) were used for the study. The sheet was mechanically cut into coupons with dimension 5cm x 4cm. The coupons were degreased by washing in absolute ethanol, dried in acetone and stored in moisture free desiccators before use. All reagents used for the study were analar grade and double distilled water was used for their preparation. The concentrations of H_2SO_4 used for weight loss was 1M. The inhibitor, (*Vernonia Amygdalina)* was gotten from Nkwe Town, Awgu L.G.A., Enugu State, Nigeria.

I. Preparation of Inhibitor

Extraction of Plants

Samples of Vernonia Amygdalina was dried, ground and soaked in a solution of ethanol. After 48 hours, the sample was cooled and filtered. The filtrate was subjected to evaporation (to leave the sample free of the ethanol) using a rotary evaporator. The stock solution of the extract so obtained was used to prepare 0.1g/L, 0.2g/L, 0.3g/L, 0.4g/L and 0.5g/L in 1M of H_2SO_4.

II. Preparation of Reagents

The concentrations of the various reagents used for this study were prepared using recommended methods. For weight loss methods, the concentrations of tetraoxosulphate (VI) acid used was 1M. The concentrations of the inhibitor were 0.1g/L, 0.2g/L, 0.3g/L, 0.4g/L and 0.5g/L ethanol extract of Verno*nia Amygdalina*. Each of these solutions was dissolved in 1M H_2SO_4 for use in weight loss method.

2.2. Corrosion Monitoring

I. Weight Loss Method

A previously weighed metal (mild steel) was completely immersed in 250ml of the test solution (different concentrations of inhibitors) in an open beaker. The beaker was inserted into a water bath maintained at a temperature of 303°K. Similar experiments were repeated at 313, 323 and 333°K. In each case, the weight of the sample before immersion was measured using Scaltec high precision balance (Model SPB31) After every 24hours, each sample was removed from the test solution, washed in a solution of NaOH containing zinc dust and dried in acetone before re-weighing. The difference in weight for a period of 120h was taken as total weight loss. The inhibition efficiency (%η) for each inhibitor was calculated using the formula;

$$\%\eta \quad = \quad 1 - \frac{W_1}{W_2} \text{ x } 100 \qquad (1)$$

where W_1 and W_2 are the weight losses (g/dm^3) for mild steel in the presence and absence of inhibitor in H_2SO_4 solution respectively. The degree of surface coverage θ is given by the Eq.(2): $\theta = 1 - \frac{W_1}{W_2}$ (2)

The corrosion rates for mild steel corrosion in different concentrations of the acid and other media have been determined for 120h immersion period from weight loss using Eq.3 [43]

$$\text{Corrosion rate (mpy)} = \frac{534W}{DAT} \quad (3)$$

Where W = weight loss (mg); D = density of specimen (g/cm^3), A = area of specimen (square inches) and T = period of immersion (hour).

II. Fourier Transformation Infra Red Analysis

FTIR analysis of the corrosion product of mild steel in 1M H_2SO_4 and in the presence of Vernonia Amygdalina were carried out at National Research Institute of Chemical Technology (NARICT), Zaria, Kaduna State, Nigeria using Fourier Transform Infra Red spectrophotometer

III. Results And Discussions

1.2. Results

Table 1 presents values of corrosion rates of mild steel at various temperatures in the absence and presence of various concentrations of ethanol extract of Vernonia Amygdalina. Values of inhibition efficiency of ethanol extract of Vernonia Amygdalina calculated from Eq. (1) are also presented in Table 1.

Table 1: Corrosion Rate and Inhibition Efficiency of Various Concentrations of Vernonia Amygdalina for Mild Steel

C (g/L)	Corrosion rate x 100000				Inhibition efficiency (%)			
	303 K	**313 K**	**323 K**	**333 K**	**303 K**	**313 K**	**323 K**	**333 K**
Blank	12.02	26.27	25.80	33.39				
0.1	1.93	4.97	4.88	7.53	83.91	81.09	81.08	77.45
0.2	1.76	4.64	4.55	7.32	85.40	82.36	82.34	78.08
0.3	1.64	4.55	4.46	7.08	86.39	82.71	82.68	78.80
0.4	1.49	4.52	4.43	6.88	87.62	82.83	82.79	79.40
0.5	1.31	3.94	3.87	6.79	89.11	85.00	84.90	79.66

The results indicated that in the presence of ethanol extract of *Vernonia Amygdalina*, the corrosion rate is decreased, even as the concentration of the extract increases. The trend for the decrease is presented graphically in Fig. 1.

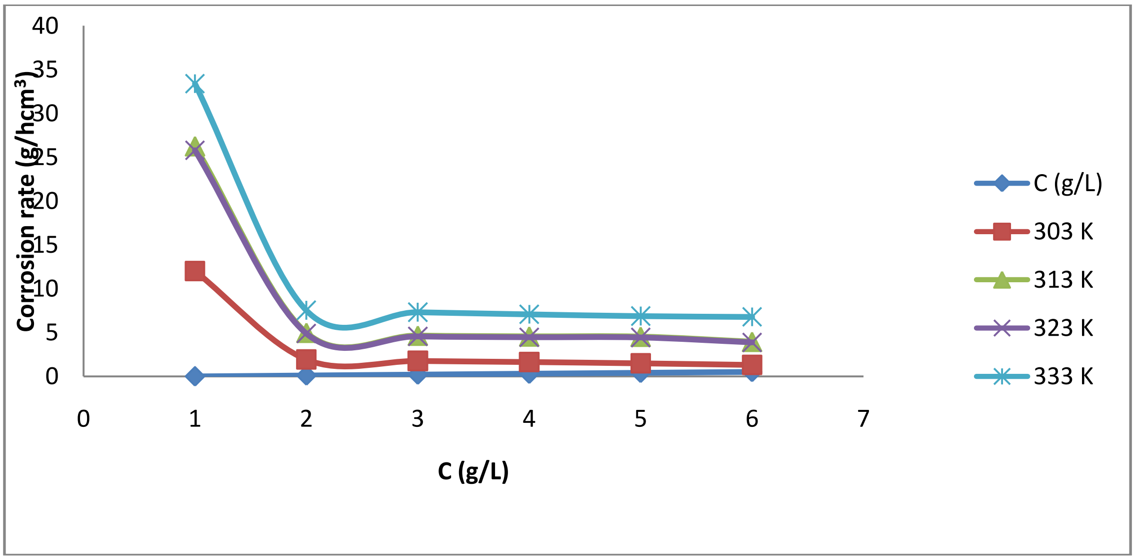

Fig.1: Variation of Corrosion Rate of Mild Steel with Concentration of *Vernonia Amygdalina* at Various Temperatures

The plot generally reveals that the decrease in corrosion rate varies with the concentration of ethanol extract of Vernonia Amygdalina. From the results obtained (Table 1), the inhibition efficiency seems to increase with concentration (Fig. 2) but decreases with temperature indicating that the mechanism of inhibition of the corrosion of mild steel in solution of H_2SO_4 is physical adsorption.

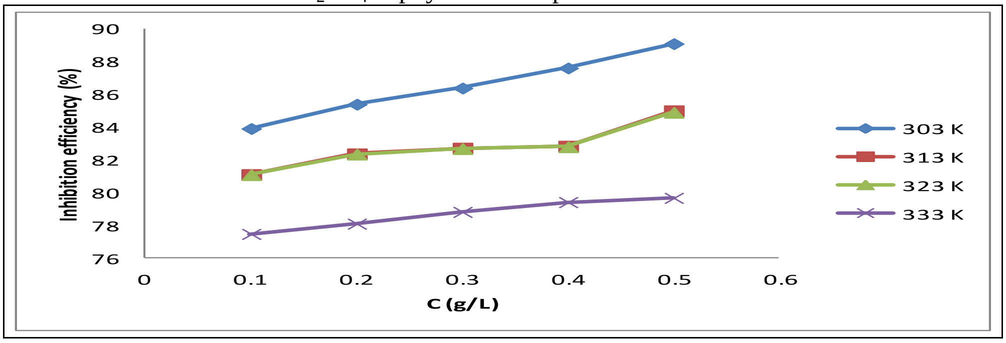

Fig. 2: Variation of the Inhibition Efficiency of Vernonia Amygdalina for the Corrosion of Mild Steel in 1 M H_2SO_4 at Various Temperatures

A physical adsorption mechanism is characterized with a decrease in inhibition efficiency with temperature as opposed to chemical adsorption mechanism, where inhibition efficiency is expected to increase with increase in temperature [12, 13]

1.3. Effect of Temperature

Temperature affects the rate of any chemical reaction including corrosion reaction. The effect of temperature on the rate of corrosion of mild steel can be modeled using the Arrhenius equation, which can be expressed as follows [14],

$$CR = Aexp\left(\frac{-E_a}{RT}\right) \qquad (4)$$

Where CR is the corrosion rate of mild steel in solution of H_2SO_4, A is the Arrhenius constant or pre-exponential factor, E_a is the minimum energy needed for the corrosion reaction to start (i.e activation energy), R is the universal gas constant and T is the temperature. Logarithm of both sides of Eq.(4) yields Eq.(5) as expressed below,

$$\log(CR) = \ln(A) - \frac{-E_a}{RT} \qquad (5)$$

The implication of equation (4.2) is that a plot of log(CR) versus 1/T should be linear with slope equals to $-E_a/R$ and intercept equals to ln(A).

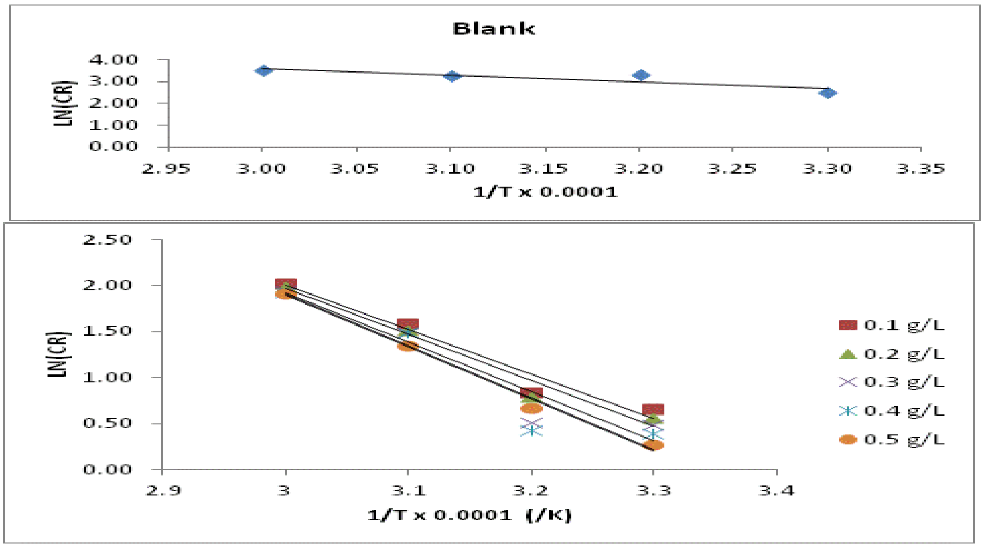

Fig. 3: Arrhenius Plot for the Corrosion of Mild Steel in the Absence and Presence of Vernonia Amygdalina as an Inhibitor at Various temperatures.

Fig. 3 shows the Arrhenius plots for the corrosion of mild steel in the absence and presence of various concentrations of ethanol extract of *Vernonia Amygdalina*. Values of Arrhenius parameters calculated from the plots are presented in Table 2.

Table 2: Arrhenius Parameters for the Inhibition of the Corrosion of Mild Steel by Various Concentrations of Vernonia Amygdalina

C (g/L)	Slope	LN(A)	E_a (J/mol)	R^2
Blank	3.4647	13.938	28.81	0.9922
0.1	4.5522	15.684	37.85	0.9997
0.2	4.7307	16.243	39.33	1.0000
0.3	4.8924	16.648	40.68	0.9996
0.4	5.1493	17.406	42.81	0.9975
0.5	5.4749	18.334	45.52	0.9999

From the results obtained, it is evident that excellent degree of linearity (R^2 ranged from 0.9922 to 1.000) were obtained in all cases indicating the application of the Arrhenius model to the inhibited and uninhibited corrosion reaction of mild steel. Value of the activation energy for the blank was 28.81 J/mol. In the presence of the inhibitor, E_a values ranged from 37.85 to 45.52 J/mol and were found to increase with increase in concentration of the inhibitor**.** Therefore, the corrosion of mild steel is retarded by the presence of ethanol extract of Vernonia Amygdalina and the ease of adsorption of the inhibitor on the surface of the metal increases with increasing concentration. It has been found for most corrosion reaction that values of activation energy less than 80 kJ/mol is associated with the mechanism of physical adsorption while those more than 80 kJ/mol points toward the mechanism of chemical adsorption. Hence the mechanism of adsorption of ethanol extract of Vernonia Amygdalina on the surface of mild steel is consistent with charge transfer from charged inhibitor to charged metal surface, which favors physical adsorption [15].

1.4. Thermodynamics/Adsorption Considerations

Thermodynamic parameters (including enthalpy and entropy of adsorption) for the adsorption of ethanol extract of *Vernonia Amygdalina* on the surface of mild steel was investigated using Eyring transition state equation, which can be expressed as follows[15]

$$ln\left(\frac{CR}{T}\right) = ln\left(\frac{R}{Nh}\right) + \frac{\Delta S^0_{ads}}{R} - \frac{\Delta H^0_{ads}}{RT} \qquad (6)$$

From Eq. (6), a plot of ln(CR/T) versus 1/T should be linear with slope and intercept equal to $\frac{\Delta H^0_{ads}}{R}$ and $ln\left(\frac{R}{Nh}\right) + \frac{\Delta S^0_{ads}}{R}$ respectively.

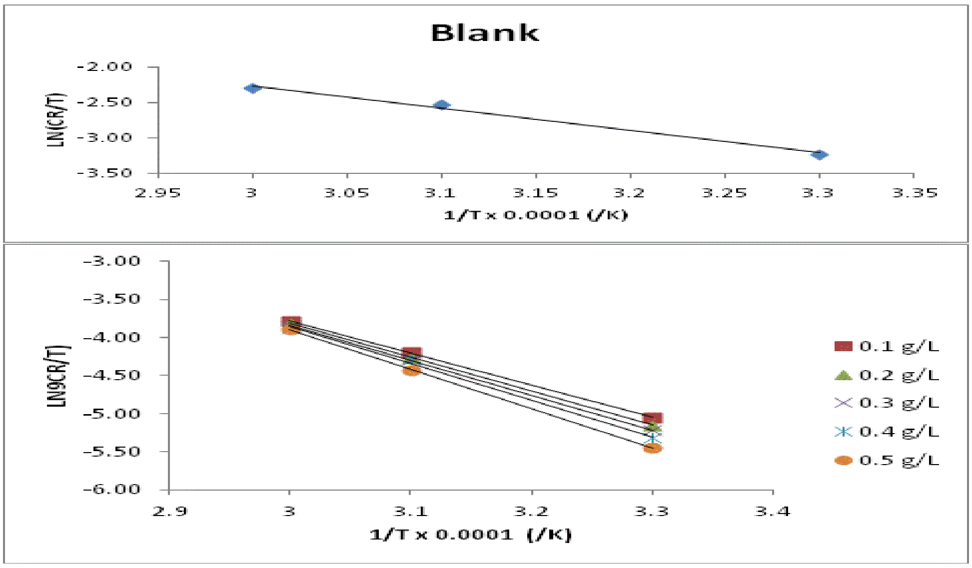

Fig. 4: Transition State Plot for the Inhibition of the Corrosion of Mild Steel in 1 M H_2SO_4 by Various Concentrations of Vernonia Amygdalina

Fig. 4 shows the transition state plot for the corrosion of mild steel in 1 M H_2SO_4 containing various concentrations of ethanol extract of Vernonia Amygdaiina. Thermodynamic parameters deduced from the plots are presented in Table 3.

Table 3: Transition State Parameters for the Inhibition of the Corrosion of mild Steel by Various Concentrations of Vernonia Amygdalina

C	Slope	Intercept	ΔS^0_{ads} (J/mol)	ΔH^0_{ads} (J/mol)	R^2
Blank	3.1493	7.1829	257.26	-26.18	0.9908
0.1	4.2368	8.9294	271.78	-35.22	0.9997
0.2	4.4353	9.4870	276.42	-36.88	1.0000
0.3	4.5780	9.8938	279.80	-38.06	1.0000
0.4	4.8339	10.6520	286.10	-40.19	0.9972
0.5	5.1595	11.5800	293.82	-42.90	0.9999

From the results obtained, R^2 values are very close to unity indicating the application of the Transition state model to the studied corrosion reaction. Values of standard enthalpy change deduced from the plots were found to be negative and ranged from 35.22 to 42.90 J/mol. These values are greater than that of the blank (enthalpy change for the blank = -26.18 J/mol) and tend to increase with increase in concentration of ethanol extract of *Vernonia Amygdalina*. Therefore, the corrosion of mild steel in H_2SO_4 solution is strongly retarded by ethanol extract of *Vernonia Amygdalina* and that the ease of adsorption of the inhibitor on the metal surface increases with increasing concentration. On the other hand, values of entropy change were positive indicating that for the adsorption of ethanol extract of *Vernonia Amygdalina* to be spontaneous, the enthalpy values should be negative as found in the present study.

The adsorption characteristics of ethanol extract of *Vernonia Amygdalina* for mild steel was investigated using adsorption isotherms including Langmuir, Fruendlich, Flory-Huggins, El awardy et al., Bockris-Swinkel and Temkin isotherms. Fitness of the data obtained for the degree of surface coverage reveals that Langmuir model best described the adsorption ethanol extract of *Vernonia Amygdalina* on the surface of mid steel. The Langmuir adsorption model can be expressed as follows [16].

$$log\left(\frac{C}{\theta}\right) \quad = \quad log b_{ads} - logC \qquad (7)$$

where C is the concentration of ethanol extract of *Vernonia Amygdalina* in the bulk electrolyte, θ is the degree of surface coverage of the inhibitor and b_{ads} is the equilibrium constant of adsorption, which is related to the standard free energy of adsorption according to equation (4.5)[17].

$$b_{ads} \quad = -\frac{1}{55.5} exp\left(\frac{\Delta G^0_{ads}}{RT}\right) \qquad (8)$$

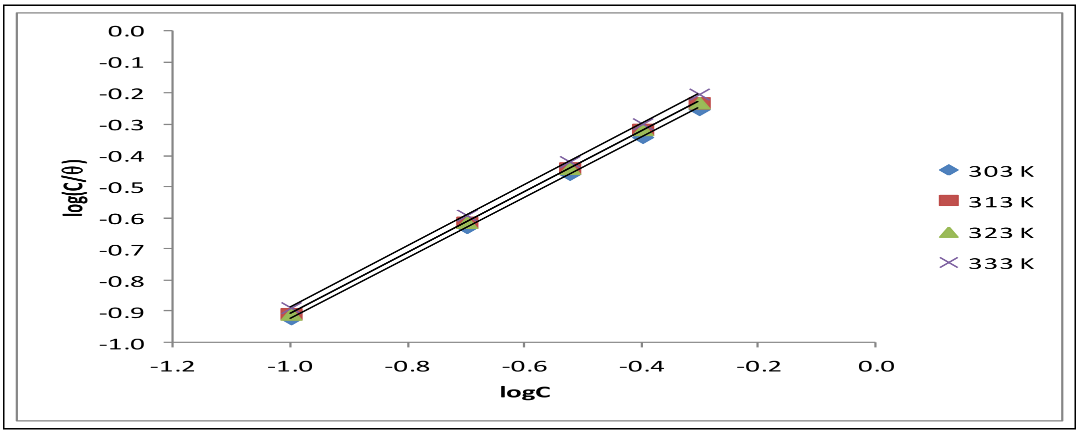

Fig. 5: Langmuir Isotherm for the Adsorption of Ethanol Extract of *Vernonia Amygdalina* on the Surface of Mild Steel.

Fig. 5 shows the Langmuir plots developed through Eq.(7), while adsorption parameters (including values ofΔG^0_{ads}, calculated from Eq.(8)) deduced from the Langmuir plots are presented in Table 4. Slope values are seen to proximate unity, which suggest that there is little or no interaction between the inhibitor and the metal. Excellent correlations obtained for the different temperatures studied confirm the application of Langmuir isotherm to the adsorption of ethanol extract of *Vernonia Amygdalina* on mild steel. Calculated values of the free energy are within the range of values expected for the mechanism of physical adsorption hence the adsorption of ethanol extract of *Vernonia Amygdalina* on the surface of mild steel is consistent with the mechanism of physical adsorption [17].

Table 4: Langmuir Parameters for the Adsorption of Ethanol Extract of Vernonia Amygdalina

T (K)	Slope	$logb_{ads}$	ΔG^0_{ads} $(kJ/mol$	R^2
303	0.9646	0.0425	-10.36	0.9999
313	0.9762	0.0681	-10.51	0.9998
323	0.9767	0.0686	-10.52	0.9999
333	0.9819	0.0936	-10.66	1.0000

1.5. FTIR Study

The interpretation of infrared spectra involves the correlation of absorption bands in the spectrum of an unknown compound with the known absorption frequencies for types of bonds. Every molecule has its own characteristic spectrum. The bands that appear depend on the types of bonds and the structure of the molecule.

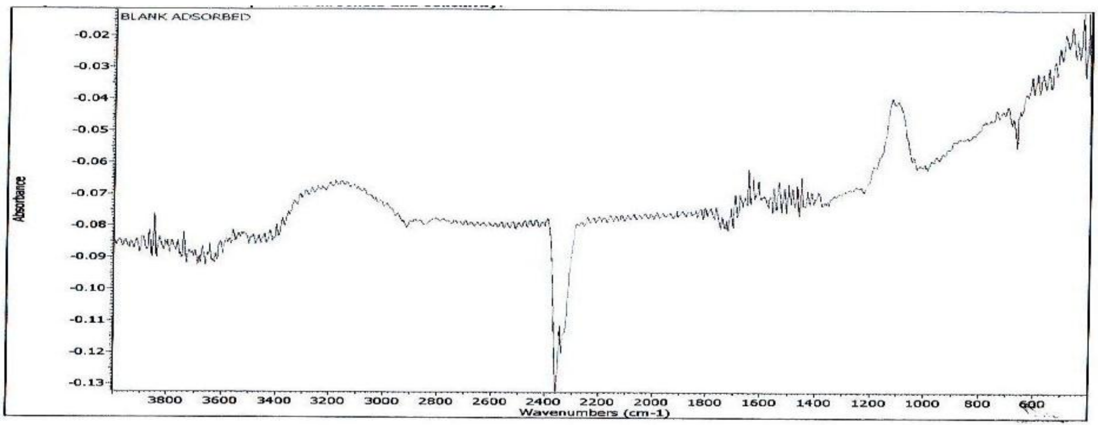

Fig. 6: IR Spectrum of the Corrosion Product of Mild Steel in 1 M H_2SO_4 (without inhibitor)

Fig. 6 shows IR spectrum of the corrosion product of mild steel without an inhibitor. It is evident that there is no serious adsorption without the inhibitor but Table 5 presents frequencies of adsorption of IR by ethanol extract of *Vernonia Amygdalina*, with and without corrodent.

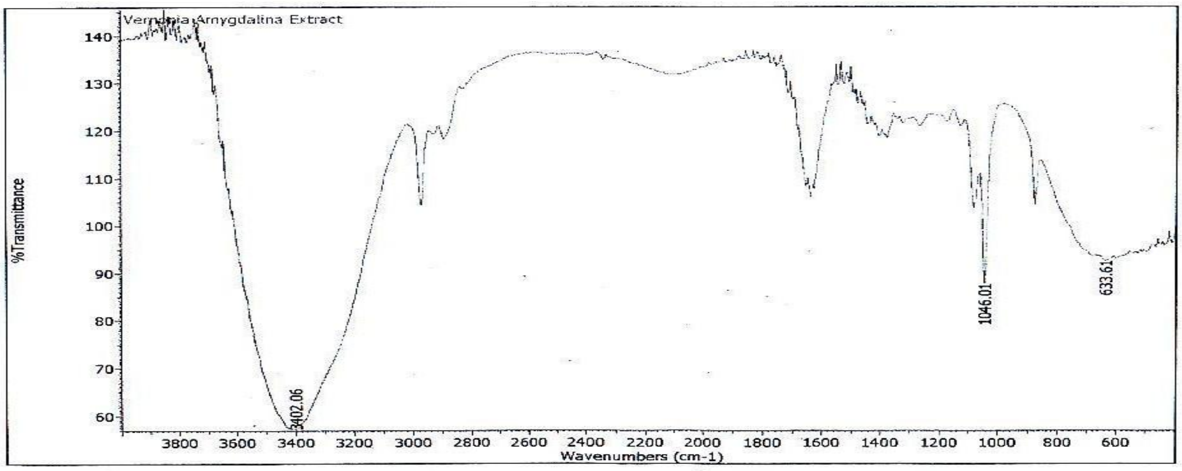

Fig. 7: IR Spectrum of Ethanol Extract of *Vernonia Amygdalina*

Fig. 7 shows IR spectrum of the corrosion product of ethanol extract of *Vernonia* Amygdalina while Fig. 8 shows the IR spectrum of the corrosion product of mild steel when 0.5 g/L of ethanol extract of *Vernonia Amygdalina* was used as an inhibitor.

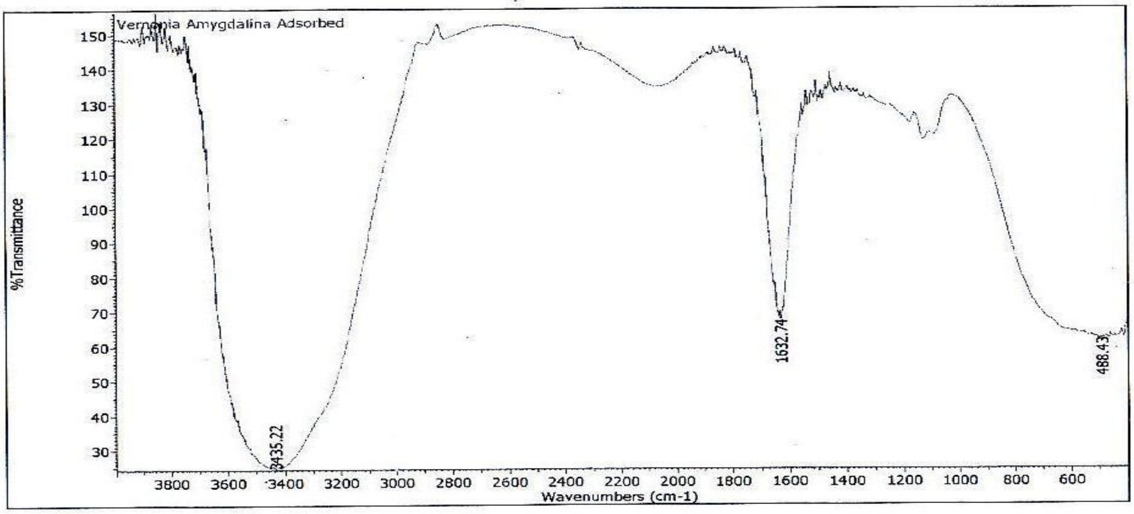

Fig. 8: IR Spectrum of the Corrosion Product in the Presence of Ethanol Extract of *Vernonia Amygdalina*

From the results obtained, it is evident that the –OH stretch (3407.06 cm^{-1}) in the ethanol extract is shifted to 3435.22cm^{-1} and the C=O stretch (1046.01cm^{-1}) is shifted to 1632.74cm^{-1} while C-H bend (633.61cm^{-1}) is absent in the spectrum (Fig. 3) indicating that there is interaction between the extract and the mild steel surface and that the C-H bend must have been involved in bonding [18]. Similar spectral information for biologically active c-alkylated flavonoids from *Vernonia Amygdalina* was also reported by Leelavathi *et al* [19]. They have isolated the following c-alkylated flavonoids such as 5,7-Dihydroxy -3' –(4" –acetoxy -3" methylbutyl) – 3,6,4' –trimethoxyflavone, and 5,7 – Dihdroxy -3' –(3-hydroxymethylbutyl)- 3,6,4' – trimethoxyflavone from the arial parts of *Vernonia Amygdalina.* From the FTIR studies and the reported phytochemical constituents by Leelavathi *et al*, it is confirmed that flavonoids are the main components present in *Vernonia Amygdalina.*

Table 5: Frequencies and Peaks of Adsorption of IR by Ethanol Extract of *Vernpnia Amygdalina*

Ethanol extract			Extract adsorbed on mild steel		
Frequency (cm^{-1})	**Height (cm)**	**Assignment**	**Frequency (cm^{-1})**		**Assignment**
3402.06	56.880	-OH stretch	3435.22		-OH stretch
1046.01	87.459	C=O stretch	1632.74		-C=O stretch
633.61	92.300	C-H bend	488.43		

IV. Conclusion

The aim of the present study was to investigate the effects of corrosion inhibition efficiency of ethanol extract of *Vernonia Amygdalina* for the corrosion of mild steel at various temperatures. In order to achieve this, weight loss (gravimetric) and FTIR methods of studying corrosion were used. From the results and findings of the study, it can be concluded that ethanol extract of *Vernonia Amygdalina* is an adsorption inhibitor for the corrosion of mild steel in acidic medium. The inhibitor modified the surface of the metal by been adsorbed on it through the mechanism of physical adsorption. The adsorption of the inhibitor on the surface of the metal forms protective layer, which inhibited corrosion reaction. The adsorption of ethanol extract of *Vernonia Amygdalina* on the surface of mild steel is best described by the Langmuir adsorption model. Thermodynamic considerations revealed that the adsorption is exothermic and spontaneous. In view of their good inhibition efficiency, the use of ethanol extract of *Vernonia Amygdalina* as corrosion inhibitor for mild steel is advocated in this paper. However, future study could be directed towards the adoptions of electrochemical methods in order to elucidate the mechanism of adsorption.

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