

## Inhibitory Action of *Nicotiana tabacum* Extracts on the Corrosion of Mild Steel in HCl: Adsorption and Thermodynamics Study

Olasehinde, E. F.<sup>1</sup>, Olusegun S. J.<sup>2</sup>, Adesina, A. S.<sup>1</sup>, Omogbehin, S. A.<sup>3</sup>, Momoh-Yahayah, H.<sup>4</sup>

<sup>1</sup>Department of Chemistry, Federal University of Technology, Akure, Nigeria

<sup>2</sup>Department of Metallurgical and Materials Engineering, Federal University of Technology, Akure, Nigeria

<sup>3</sup>Department of Science Laboratory and Technology, Waziri Umaru Federal Polytechnic, Birnin Kebbi, Nigeria

<sup>4</sup>Department of Chemistry and Centre for Agrochemical Technology, University of Agriculture, Markudi, Nigeria

[folasemb2004@yahoo.com](mailto:folasemb2004@yahoo.com)

**Abstract:** The toxic effects of synthetic corrosion inhibitors have led to the search for naturally occurring substances which are not only readily available but are also environmentally friendly. Therefore, this study investigates the inhibition efficacy of acid extract of *Nicotiana tabacum* leaves on mild steel in 1M HCl using weight loss method. Experiments are performed by varying immersion period, concentration of the inhibitor and temperature. The inhibition efficiency is markedly higher on the addition of *Nicotiana tabacum* leaves extract in acidic medium compared with those in the absence of inhibitor. The inhibition efficiency increased with an increase in inhibitor concentration but decreased with rise in temperature and exposure time. Thermodynamic studies revealed that corrosion inhibition may be due to the spontaneous physical adsorption of the plant constituents on the surface of mild steel. Experimental data fitted with the Langmuir and Temkin adsorption isotherms. Kinetic treatment of the data followed a first order reaction. Calculated half-lives increase as the concentration of the extracts increases suggesting that inhibition efficiency increases with increase in the concentration of the extracts. Preliminary investigation of the phytochemical constituents showed that *Nicotiana tabacum* contains tannin, flavonoid, terpenoid and some other compounds in trace constituents.

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### 1. Introduction

The toxic effects of synthetic corrosion inhibitors have led to the search for naturally occurring substances which are not only readily available but are also environmentally friendly. It is well known that acid solutions are often used in industry for cleaning, descaling and pickling of metallic structures; processes which are normally accompanied by considerable dissolution of the metal resulting into corrosion (Saratha and Vasudha, 2010; Vijayalakshmi et al, 2011; James and Akaranta, 2009). However, a useful method to protect metals and alloys against corrosion in such an aggressive environment is the addition of organic or inorganic species to the solution in order to inhibit the corrosion reaction and hence reduce the corrosion rate. In acid media, nitrogen-based materials and their derivatives such as sulphur containing compounds, aldehydes, thioaldehydes, acetylenic compounds, and various alkaloids such as papaverine, strychnine, quine, and nicotine have been employed as inhibitors. In neutral media, benzoate, nitrite, chromate and phosphate act as good inhibitors (Singh and Quraishi, 2012; Okafor et al., 2012). These compounds function via adsorption of the molecules on the metal surface, increasing or decreasing the anodic or

cathodic reaction, decreasing the diffusion rate for reactants to the surface of the metal and/or decreasing the electrical resistance of the metal surface. The adsorption bond strength is dependent on the composition of the metal and the corrodent, the inhibitor structure and the concentration as well as the temperature (Quraishi et al., 2009; Saratha and Vasudha, 2009).

Though many synthetic compounds showed good anti-corrosive activity, most of them are highly toxic to both human beings and environment. The safety and environmental issues of corrosion inhibitors arisen in industries has always been a global concern. These inhibitors may cause reversible (temporary) or irreversible (permanent) damage to organ system viz, kidneys or liver, or to disturb a biochemical process or to disturb an enzyme system at some site in the body (Ajayi et al., 2011; Singh and Quraishi, 2012; Okafor et al., 2012).

Thus, there exists the need to develop a new class of corrosion inhibitors with low toxicity and good efficiency. The exploration of natural products of plant origin as inexpensive and ecofriendly corrosion inhibitors is an essential field of study. In addition to being environmentally friendly and ecologically acceptable, plant products are low-cost,

readily available and renewable sources of materials. The extracts from their leaves, barks, seeds, fruits and roots comprise of mixtures of organic compounds containing nitrogen, sulphur and oxygen atoms and some have been reported to function as effective inhibitors of metal and alloy corrosion in different aggressive environments (Quraishi et al., 2009; Saratha and Vasudha, 2009; Ajayi et al., 2011; Singh and Quraishi, 2012; Okafor et al., 2012; Ebenso et al., 2003; Umoren et al., 2008; James et al., 2006; Abiola et al., 2007). Adeyemi and Olubomehin (2010) assessed the influence of extracts from *Anthocleista djalensis* stem bark on the corrosion of Aluminium. Deepa Rani and Selvaraj (2010) investigated the inhibitive and adsorptive properties of *Punica granatum* extract on brass in acid medium.

*Nicotiana tabacum*, or cultivated tobacco, is a perennial herbaceous plant. It is found only on cultivation, where it is the most commonly grown of all plants in the *Nicotiana* genus, and its leaves are commercially grown in many countries to be processed into tobacco. The plant contains nicotine, which can be extracted and used as an insecticide. The dried leaves can also be used and they remain effective for 6 months after drying. The juice of the leaves can be rubbed on the body as an insect repellent. The leaves can be dried and chewed as an intoxicant. The dried leaves are also used as snuff or are smoked (Groark, 2010). Therefore, we have investigated the inhibitory action of *Nicotiana tabacum* leaves extracts on the corrosion of mild steel in acidic medium with a view to understanding the mechanism of its adsorption and the thermodynamic parameters.

## 2. Material and Methods

### Stock solution of *Musa sapientum* peel extract

The leaves of *Nicotiana tabacum* were purchased and oven dried at temperature of 95°C, grounded and sieved through a mesh 850 micron sieve. Stock solutions of the plant extract were prepared by soaking 10g each of the powdered material with 100ml of 1M HCl in a round-bottomed flask. The blank corrodent was 1M HCl solution. The solutions of the plants material were boiled at 90°C for 3 hours. The solutions were left to cool overnight, then filtered and stored. From the respective stock solutions, inhibitor test solutions were prepared in concentration of *Nicotiana tabacum* extract ranging from 0.1- 10% v/v of the 1M HCl solution.

### Specimen Preparation

Mild steel of chemical composition in percentages of Fe=98.721, C=0.181, Si=0.056, Mn=0.474, S=0.039, Ni=0.078, Cu=0.198, Al=0.124, P=0.039, Cr=0.038 and others=0.075 were used for the study. The steel was cut to form different coupons of dimensions of diameter ranging from 5.35-

5.67mm and height ranging from 18.03-21.14mm using electronic venial caliper. 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. Accurate weights of the coupons were determined using an analytical balance of 0.0001 mg accuracy.

### Weight Loss Method

After initial weighing, the specimen in triplicate were immersed in 100ml of 1M HCl solution in the absence and presence of different concentrations (0.1%, 0.5%, 1%, 1.5%, 2%, 4%, 6%, 8%, and 10% v/v) of plants extracts with the aid of glass hooks at different temperatures viz 303, 313, 323 and 333k for 6 hours. The thermostated water bath was set to the appropriate temperature and after 6 hours of immersion, the specimens were removed, washed, dried completely and their final weights were noted. From the initial and final weights of the mild steel, the weight loss, the corrosion rate (CR) ( $\text{gcm}^{-2} \text{hr}^{-1}$ ), inhibition efficiency (IE) (%), and surface coverage ( $\theta$ ) were determined using equations 1, 2 and 3 respectively (Wabane and Okafor, 2001).

$$\text{CR} = \frac{W}{At} (\text{gcm}^{-2} \text{h}^{-1}) \quad (1)$$

W is weight loss of the mild steel after time t (grams), A is the area of the mild steel coupon ( $\text{cm}^2$ ) and t is the time of immersion (hours)

$$\text{IE} (\%) = (1 - \frac{W_1}{W_2}) \times 100. \quad (2)$$

$$\theta = (1 - \frac{W_1}{W_2}) \quad (3)$$

Where  $W_1$  and  $W_2$  are the weight losses for mild steel in the presence and absence of inhibitor. All the reagents used for the study were analar grade with double distilled water.

### Chemical Analysis

Phytochemical analysis of the *Nicotiana tabacum* extract was carried out according to the method reported by Onyeka and Nwabekwe, 2007.

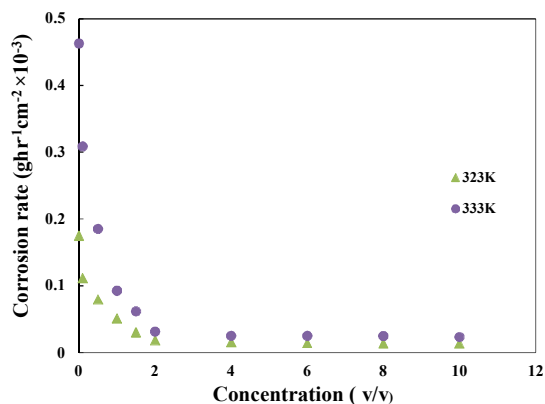
## 3. Results

### 3.1 Effect of Reaction Conditions on Corrosion Rate and Inhibition Efficiency of *Nicotiana tabacum* Extract

#### 3.1.1 Effect of Concentration on Corrosion Rate

The variation of corrosion rate of mild steel in 1M HCl in the absence and presence of various concentrations of acid extract of *Nicotiana tabacum* was studied at 323 and 333K respectively. The result (Figure 1) shows that the corrosion rate of mild steel in 1M HCl decreases with increase in the concentration of the extracts at both temperature values. This suggests that as the concentration of the

extract increases, there is an increase in the number of adsorption of the extract constituents on the surface of the mild steel which makes a barrier for mass transfer and prevents further corrosion. This result is in consonance with the findings of Wabanne and Okafor, 2001.



**Figure 1: Effect of concentration of *Nicotiana tabacum* extract on the corrosion rate of mild steel**

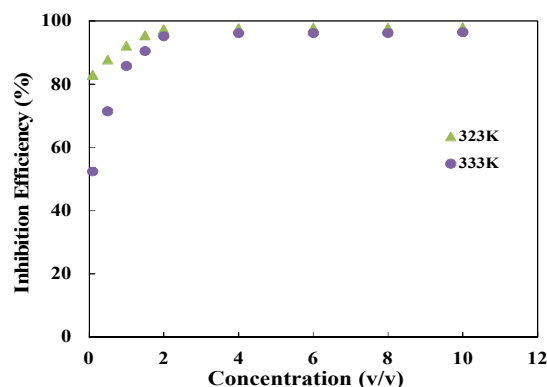
### 3.1.2 Effect of Concentration on Inhibition Efficiency

The inhibition efficiency of mild steel exposed to different concentrations of *Nicotiana tabacum* extract in 1 M HCl at 323 and 333 K is shown in Figure 2. The inhibition efficiency of the extracts was observed to increase with increasing concentration of acid extracts of the inhibitors as a result of increase in the fraction of the mild steel surface covered by the adsorbed constituents of the extract as the concentration of the plant extracts increases. The inhibition efficiency increases progressively as the concentration of the extracts increases up to about 4%, above which, further increase in extract concentration did not cause any significant change in the performance of the extracts which might indicate that the reaction of the inhibitors on the surface of the mild steel have reached the state of equilibrium. The maximum percentage inhibition of 99.47% was recorded at the highest concentration studied at 323K.

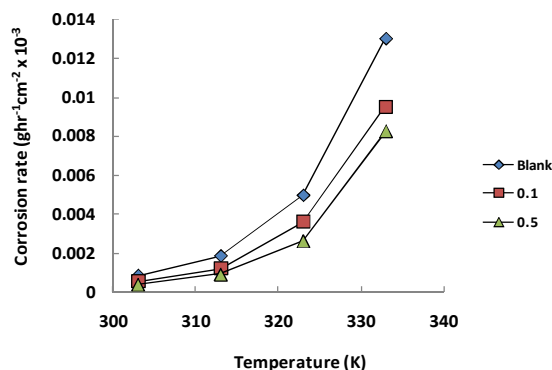
### 3.1.3 Effect of temperature on corrosion rate

The effect of temperature on the corrosion rate of mild steel in free acid and in the presence of different concentrations of the inhibitor (plant extract) was studied in the temperature range of 303K to 333K as shown in Figure 3. It was found that the rates of corrosion of mild steel in free acid solution and in the presence of different concentrations of the inhibitor increase with increase in temperature. This is expected because as temperature increases, the rate of corrosion of mild steel also increases as a result of

increase in the average kinetic energy of the reacting molecules. However, the corrosion rate is much decreased for inhibited acid solution than the uninhibited acid solution. The decrease in the corrosion rate for the inhibited acid solution compared to the uninhibited is as a result of the mitigating effect of the plant extract on the corrosion rate of the mild steel.



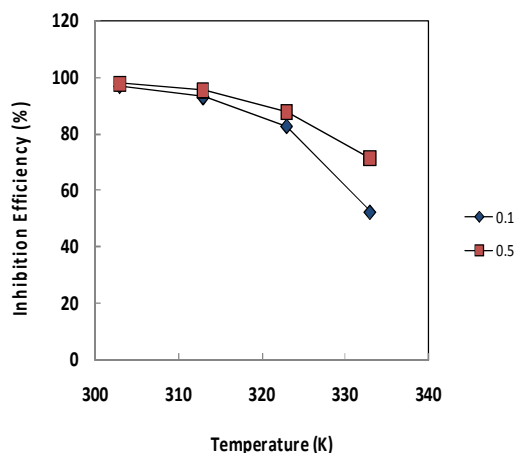
**Figure 2: Effect of concentration on the inhibition efficiency of *Nicotiana tabacum* extract**



**Figure 3: Effect of temperature on corrosion rate of mild steel**

### 3.1.4 Effect of temperature on inhibition efficiency

To evaluate the stability of adsorbed layer/film of inhibitor on mild steel surface, weight loss measurements were carried out in the range of temperature 303–333 K in the absence and presence of the extract. The results obtained are shown in Figure 4. It is evident from this figure that inhibition efficiency decreases with increasing temperature. This is due to increased rate of dissolution process of mild steel and partial desorption of the inhibitor from the metal surface with temperature (Blaedel and Meloche, 1963).



**Figure 4: Effect of temperature on the inhibition efficiency of the extract on corrosion of mild steel**

### 3.2 Adsorption Consideration

Adsorption isotherms are very important in determining the mechanism of organo-electrochemical reaction. The inhibition of the corrosion of mild steel in 1M HCl medium with addition of different concentrations of the extract can be explained by the adsorption of the components of the plant extract on the metal surface. Inhibition Efficiency (%) is directly proportional to the fraction of the surface covered by the adsorbed molecule ( $\theta$ ). Therefore,  $\theta$  with the extract concentration specifies the adsorption isotherm that describes the system given the relationship between the coverage of interface with the adsorbed species and the concentration of species in solution. The value of the surface coverage ( $\theta$ ) at different concentrations of the inhibitors in 1M HCl solution for the extract was made to fit to various adsorption isotherms. An inhibitor is found to obey Langmuir if the plot of  $\log \frac{\theta}{1-\theta}$  vs  $\log C$  or the plot of  $\log \frac{C}{\theta}$  vs  $\log C$  is linear. Similarly, for Temkin the plot of  $\theta$  vs  $\log C$ , for BET  $\log C - \log \theta$  vs  $\theta^{3/2}$  and for Frumkin plot  $\theta$  vs  $\log C$  will be linear. In this study, Langmuir and Temkin adsorption isotherms were found to be suitable for the experimental findings. The isotherm is described by the equation (4) as given in Ismail, et al., (2011) and Adeyemi and Olubomehin, (2010).

$$\frac{C}{\theta} = 1/K_{ads} + C \quad (4)$$

Where  $C$  is the Inhibitor concentration,  $K_{ads}$  is the Adsorption equilibrium constant and  $\theta$  is the Surface coverage.

Taking logarithm of equation 4 above yields equation (5)

$$\log \left( \frac{C}{\theta} \right) = \log C - \log k_{ads} \quad (5)$$

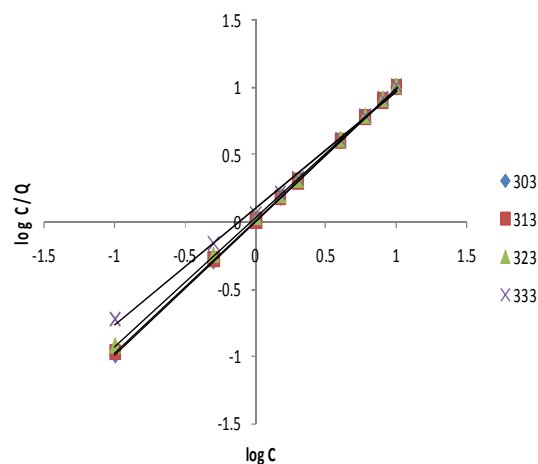
The plot of  $\log \left( \frac{C}{\theta} \right)$  versus  $\log C$  was linear with the intercept equal to  $-\log K_{ads}$  for the extract (see Fig. 5). The estimated values for  $K_{ads}$  and correlation coefficient ( $r^2$ ) for the extract at various temperatures are shown in the Table 1:

**Table 1: Changes in Adsorption Equilibrium constant ( $k_{ads}$ ) at different Temperatures**

Temperature	$k_{ads}$	$r^2$
303	0.9844	0.9961
313	0.9687	0.9989
323	0.9165	1.0000
333	0.7859	1.0000

The plot of  $\theta$  vs  $\log C$  was also linear (see Fig. 6). The plots support the assertion that the mechanism of corrosion inhibition is due to the formation and maintenance of a protective film on the metal surface and that the additive covers both the anodic and cathodic sites through uniform adsorption (Adeyemi and Olubomehin, 2010). The fit of the experimental data to these isotherms provide evidence for the role of adsorption in the observed inhibitive effect of the *Nicotiana tabacum*.

$$\ln (CR_2/CR_1) = Ea/ R (1/T_1 - 1/T_2)$$



**Figure 5: Langmuir isotherm**

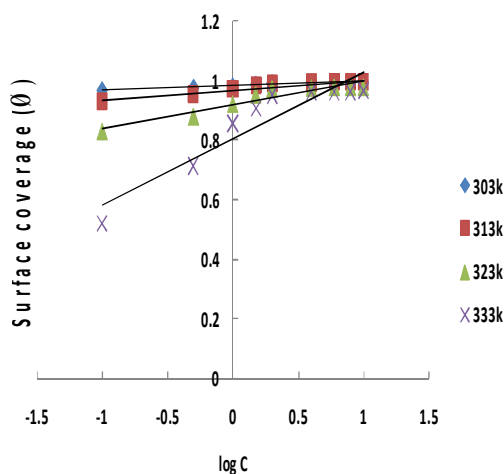


Figure 6: Temkin isotherm

### 3.3 Thermodynamic Study

#### 3.3.1 Determination of Activation Energy

Analysis of the temperature dependence of inhibition efficiency as well as comparison of corrosion activation energies in the presence and absence of inhibitor gives some insight into the possible mechanism of inhibitor adsorption. A decrease in inhibition efficiency with rise in temperature, with analogous increase in corrosion activation energy in the presence of inhibitor compared to its absence, is frequently interpreted as being suggestive of formation of an adsorption film of physical (electrostatic) nature (Blaedel and Meloche, 1963). Thus the apparent activation energy ( $E_a$ ) for the corrosion process in the absence and presence of the inhibitor was evaluated from Arrhenius equation (Rani and Selvaraj, 2010).

$$\ln \left( \frac{CR_2}{CR_1} \right) = \frac{E_a}{R} \left( \frac{1}{T_1} - \frac{1}{T_2} \right) \quad (6)$$

Where  $CR_1$  and  $CR_2$  are the corrosion rates at temperature  $T_1$  and  $T_2$  respectively,  $E_a$  is the apparent activation energy,  $R$  is the molar gas constant.

The estimated values of  $E_a$  for mild steel corrosion in the presence of *Nicotiana tabacum* extract in 1 M HCl are listed in the Table 2. The data shows that the activation energy ( $E_a$ ) of the corrosion in mild steel in 1M HCl solution in the presence of extract is higher than that in the free acid solution. Activation energy  $E_a$  was found to be  $59.58 \text{ KJmol}^{-1}$  for 1M HCl and increases to  $67.78 \text{ KJmol}^{-1}$  in the presence of *Nicotiana tabacum* extract at 1 % (v/v) concentration which shows that the adsorbed organic matter has provided a physical barrier to the change and mass transfer, leading to reduction in corrosion

rate (Abiola et al., 2007; Ebenso, 2003). It has been reported earlier that when the values of  $E_a > 80 \text{ kJ/mol}$  indicates chemical adsorption whereas  $E_a < 80 \text{ kJ/mol}$  infers physical adsorption (Ismail et al., 2011; Adeyemi and Olubomehin, 2010). On the basis of the experimentally determined activation energy value of  $E_a < 80 \text{ kJ/mol}$  in this study, we propose that the additive is physically adsorbed on the coupons. Therefore, it is plausible that a multilayer protective coverage on the entire mild steel surface was obtained.

Table 2: The Activation Energy values with the various concentrations of the extract

Extract Concentration	$E_a$ (Kj/mol)
Blank	59.58
0.1	66.34
0.5	67.73
1.0	67.78

#### 3.3.2 Determination of Enthalpy and Entropy

Other thermodynamic parameters such as enthalpy ( $\Delta H$ ) and entropy ( $\Delta S$ ) of activation of corrosion process may be evaluated from the effect of temperature. The enthalpy ( $\Delta H^\circ$ ) and entropy ( $\Delta S^\circ$ ) of activation of corrosion process was calculated from the equation:

$$\log \frac{CR}{T} = \log \left( \frac{R}{nh} \right) + \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303RT} \quad (7)$$

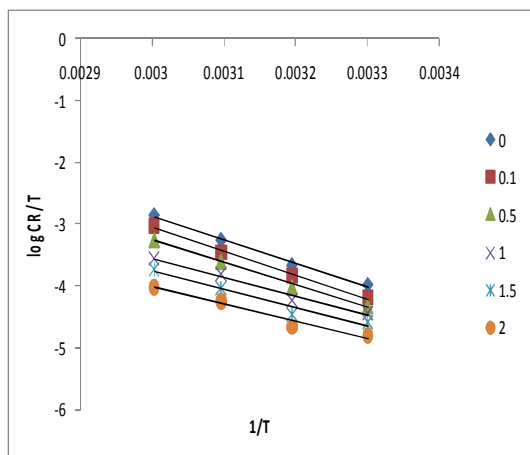
Where  $CR$  is the Corrosion Rate at Temperature  $T$ ,  $R$  is the molar gas constant,  $n$  is Avogadro's constant, and  $h$  is the Planck's constant. A plot of  $\log \frac{CR}{T}$  vs  $\frac{1}{T}$  is a straight line graph (see Fig. 7)

with a slope of  $\left( -\frac{\Delta H}{2.303RT} \right)$  and an intercept of  $\left( \log \left( \frac{R}{nh} \right) + \frac{\Delta S}{2.303R} \right)$  from which the values of  $\Delta H$  and  $\Delta S$  were calculated as discussed by (Abiola et al., 2003; Ebenso et al., 2007; James, et al., 2006).

The results presented in Table 3 shows that the enthalpy of activation values are all positive for *Nicotiana tabacum* which reflects the endothermic nature of the mild steel dissolution process. Also, the entropies of activation energy were positive for the extract, indicating that the activation complex represents association steps and that the reaction was spontaneous and feasible. These results were in excellent agreement with the reports of previous work by (Abiola et al., 2007).

**Table 3: Enthalpy and Entropy of the reaction with various concentrations of the extracts.**

Extract Concentration	Enthalpy (kJ/mol)	Entropy (kJ/mol/k)
Blank	73.5899	31.9112
0.1	74.4008	32.8834
0.5	69.5040	51.5077
1.0	59.3929	87.3511
1.5	57.6304	96.6034
2.0	52.8009	116.2374



**Figure 7: Plot of  $\log \frac{CR}{T}$  vs  $\frac{1}{T}$**

**3.3.3 Determination of Free Energy**

The standard free energy of adsorption,  $\Delta G^{\circ}_{ads}$ , which can characterize the interaction of adsorption molecules and metal surface, was calculated by equation (8). The values of  $\Delta G^{\circ}_{ads}$  obtained are presented in Table 4. The negative values of  $\Delta G^{\circ}_{ads}$  ensure the spontaneity of adsorption process and stability of the adsorbed layer on the mild steel surface. Generally, the values of  $\Delta G^{\circ}_{ads}$  around -20 kJ/mol or lower are consistent with physisorption, while those around -40kJ/mol or higher involve chemisorptions (Umoren et al., 2009; Ebenso et al. 2003). As shown in the Table, results obtained indicate that the values of  $\Delta G^{\circ}_{ads}$  are negative in all cases and are less than 20 kJ/mol (~ 10 kJ/mol). This is consistent with literature survey and therefore authenticates physical adsorption. This implies that the plant extracts adheres on the surface of the corroding system and so gives a very strong inhibitor.

$$\Delta G^{\circ}_{ads} = -2.303RT \log (55.5K_{ads}) \tag{8}$$

**Table 4: The Free Energy of adsorptions at various temperatures**

Temperature	$\Delta G$ (kJ/mol)
303	-10.0801
313	-10.3708
323	-10.5535
333	-10.4544

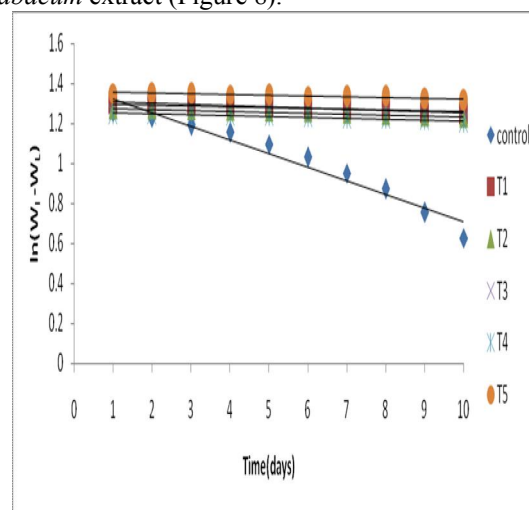
**3.4. Kinetic Study**

**3.4.1. Determination of rate constant and Half-life**

The corrosion reaction is a heterogeneous reaction which is composed of anodic and cathodic reactions at the same or different rate. It is on this basis that kinetic analysis of the data is considered necessary. In this present study, the initial weight of mild steel coupon is designated  $W_i$  while the weight after time  $t$ ,  $W_L$  hence the weight change after time  $t$ ,  $(W_i - W_L)$ . The rate constant was calculated as shown in earlier report (James et al, 2006).

$$\ln (W_i - W_L) = -k_1 t + \ln W_L \tag{9}$$

The plots of  $\ln (W_i - W_L)$  against time (in days) at 303K was studied which showed a linear variation and slope  $k_1$  which confirms a first order reaction kinetics with respect to the corrosion of mild steel in 1 M HCl solutions in the presence of the *Nicotiana tabacum* extract (Figure 8).



**(Where T<sub>1</sub>-T<sub>5</sub> are concentrations at 2, 4, 6, 8 and 10 % v/v respectively)**

**Figure 8: Variation of  $\ln (W_i - W_L)$  with time (days) for mild steel coupons in 1M HCl solution containing *Nicotiana tabacum* at 303K.**

**(ii) Half Life,  $t_{1/2}$**

The half-life ( $t_{1/2}$ ) was calculated from the relation below as shown in Table 5:

$$t_{1/2} = \frac{0.693}{K_1} \tag{10}$$

The increase in half-life ( $t_{1/2}$ ) shown when the *Nicotiana tabacum* extracts were present further supports the inhibition of mild steel in 1M HCl by the additives. As the half-life increases, the corrosion rate decreases which is an indication that more protection of the metals by the *Nicotiana tabacum* extracts has been established. As discussed earlier, the activation energy which is in the range of 59.58kJ/mol to 67.78 kJ/mol is in an indication that the additive is physically adsorbed on the metal coupons (James et al., 2006).

**Table 5: Half-life parameters at various concentrations**

Extract Concentration	Rate Constant ( $\text{day}^{-1}$ ) $\times 10^{-3}$	Half life (days)
Blank	0.06818	10.16427
0.1	0.00623	111.2360
0.5	0.00469	147.7612
1.0	0.00445	155.7303
1.5	0.00435	159.3103
2.0	0.0039	177.6923

### 5.0 Phytochemical Constituents

The major phytochemical constituents present in *Nicotiana tabacum* are listed in Table 6. As earlier reported (Onyeka and Nwabekwe, 2007), these compounds are easily hydrolysable and the compounds can be adsorbed on the metal surface via the lone pair of electrons present on their oxygen atoms (i.e. they contain multifunctional group) which make a barrier for charge and mass transfer leading to decrease the interaction of the metal with the corrosive environment. As a result, the corrosion rate of the metal was decreased. The formation of film layer essentially blocks the discharge of  $\text{H}^+$  and dissolution of the metal ions. Due to electrostatic interaction, the protonated constituent's molecules are adsorbed (physisorption) and high inhibition is expected. Acid pickling inhibitors containing organic N, S and OH groups behave similarly in inhibiting the corrosion of mild steel.

**Table 6: The Phytochemical constituents of *Nicotiana tabacum* extract**

Phytochemical	<i>Nicotiana tabacum</i>
Alkaloid	+
Saponin	-
Tannin	+
Flavonoid	+
Anthraquinone	-
Terpenoid	+

### 4. Conclusion

- (1) The acid extract of *Nicotiana tabacum* acts as good and efficient inhibitor for the corrosion of mild steel in hydrochloric acid medium.
- (2) Inhibition Efficiency (%) increases with inhibitor concentration and maximum inhibition efficiency for the extract was found to be 99.66% at the optimum concentration of 10%v/v at 303K.
- (3) The adsorption of different concentrations of the plant extract on the surface of the mild steel in 1M HCl acid followed both Langmuir and Temkin adsorption isotherm.
- (4) The effect of temperature revealed physical adsorption for the inhibition action of the plant extract.
- (5) The negative sign of the Free Energy of adsorption indicates that the adsorption of the inhibitors on the mild steel surface was a spontaneous process and was found to be physisorption.
- (6) The positive values of enthalpy of adsorption ( $\Delta H$ ) suggest that the reaction of the adsorption of the inhibitors on the metal surface is an endothermic reaction hence increase in the temperature of the medium will decrease the inhibition efficiency.
- (7) The positive values of entropy of adsorption indicate that the reaction was spontaneous and feasible.
- (8) Activation energy  $E_a$  was found to be  $59.58\text{KJmol}^{-1}$  for 1M HCl and increases to  $67.78\text{KJmol}^{-1}$  in the presence of *Nicotiana tabacum* extract at 0.1v/v which shows that the adsorbed organic matter has provided a physical barrier to charge and mass transfer, leading to reduction in the rate of corrosion.
- (9) The mechanism of the physical adsorption was proposed and a first order type of reaction is obtained from the kinetic treatment of the data.

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### Corresponding Author:

Dr. Olasehinde Emmanuel F.  
Department of Chemistry  
Federal University of Technology, Akure  
Ondo State, Nigeria  
E-mail: folasemb2004@yahoo.com

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