



**EFFECT OF TEMPERATURE IN CORROSION RATE OF MILD STEEL IN ACID
MEDIA IN PRESENCE OF *ACACIA SENEGAL* EXTRACT**

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ABSTRACT

The ethanolic extract of *Acacia senegal* are tested for their effectiveness to combat corrosion rate of mild steel in 2N hydrochloric and sulfuric acid by mass loss method at different temperatures. Temperature affects the various reaction rates so any suitable range of temperature can be maintained with the help of constant temperature bath controlled automatically to the range of $\pm 1^{\circ}\text{C}$. The value of activation energy and free energy of adsorption have also been calculated to investigate the mechanism of the corrosion inhibition. The free Gibbs adsorption energy value, ΔG_{ads} of inhibitors is negative which indicates the spontaneity of adsorption process on mild steel surface. Phenomenon of physical adsorption is proposed from the activation parameters obtained.

Keywords: Corrosion, Inhibitors, *Acacia senegal*, H_2SO_4 , Mild Steel

INTRODUCTION

Non-reversible interfacial interaction of a material with its surrounding environment is called as Corrosion. Metallic Corrosion is very common but a serious problem causing considerable revenue loss throughout the world. Extenuation of corrosion requires the application of various engineering techniques and scientific knowledge on the roll of the alloying element in the reduction

of corrosion losses and application of the film forming inhibitors are well known [1].

Mild steel finds a variety of uses in most of the chemical industries due to its low cost and easily availability for fabrication of various reaction vessels, tanks and pipes etc. In alkaline medium mild steel is fairly resistant to attack by alkali because of its passiveness. However in acid medium, the metal tends to corrode. HCl, H_2SO_4 acids have been used for drilling operation,

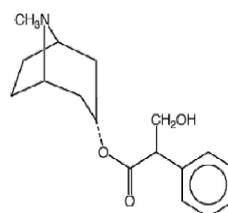
pickling baths and in descaling processes [2, 3].

The recent research oriented towards environment-friendly inhibitors. Many recent corrosion reduction methods apply coating and conversion layers which have toxic and environmentally hazardous material. There is a significant urge to discover a non-toxic/hazardous material that is also compatible with current industrial technologies. Well known as non toxic compound and due to their availability and relatively low cost, naturally substances find various applications in many fields. It has been known that natural plant products have various organic compounds e.g. alkaloids, tannin, pigments, organic and amino acids and most of them are known to have inhibitory action [4-11]. Numerous naturally occurring substances have been evaluated as potential corrosion inhibitors [12-30]. In the present work, an attempt has been made to study the influence of varying concentration of constituents and substitute extracts of *Acacia senegal* as probable acid corrosion inhibitor of mild steel by sulfuric acid.

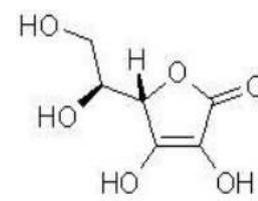
The recent discovery showed that plant extract of *Acacia senegal* (gum acacia) have antioxidant [31] and antimicrobial activity [32, 33].

Gum acacia contains neutral sugars (Rhamnose, arbinose and galactose), acids (glucuronic acid and 4-methoxy

glucuronic acid) Calcium, Magnesium, Potassium and Sodium. The molecular weight has been reported to be between 200,000 to 300,000 and as high as 600,000 [34]. Leaves contain 10-13% digestible protein and 0.12-0.14% phosphorus. The alkaloids found in *Acacia senegal* include less than 0.1% dimethyltryptamine (DMT) in leaf and N-Methyltryptamine (NMT) in other plants parts [35]. Gum acacia also contains alkaloid atropine (Figure 1) and ascorbic acid which have been found to be effective for inhibition of mild steel in acid media.



ATROPIN



ASCORBIC ACID

EXPERIMENTAL

Specimen preparation -For the mass loss determination, rectangular specimens of mild steel of size 1.5 x 2.5 x 0.25 cm were so cut from a sheet having chemical composition as C (0.14%), Mn (0.32%), S (0.05%), Tu (0.02%) and rest of mild steel. The specimens were abraded with various grades of wax coated emery paper (1/0,2/0,3/0,4/0) and successively washed with benzene, soap and distilled water and finally with acetone for cleaning. After proper drying they were weighted.

Test solution preparation

The solutions of 2N HCl and 2N H₂SO₄ were prepared using double distilled water. Analytical grade reagents were employed. The *Acacia senegal* extract was obtained from dried parts of plants, finely powdered and extracted with boiling ethanol. The solvent was distilled off, and residue treated with inorganic acid. Where the base is extracted as their soluble salts. The free bases are liberated by the addition of any base and extracted with various solvent eg. Ether, chloroform, etc. The mixture of bases thus obtained is separated by various methods into the individual compound [36].

Mass loss method

The metal coupons were than suspended with the help of glass hooks in borosil beakers containing 50 ml of corrosive electrolyte for complete immersion at different temperatures for 24 hours. After the immersion period the specimen was cleaned carefully as per champion [37] and after proper drying they were weighted again. Corrosion rate (mm/ year), inhibition efficiency (η %) and rate constant were evaluated from the mass loss results.

The inhibitor efficiency (η %) [38].

$$\eta\% = 100(\Delta M_u - \Delta M_i / \Delta M_u)$$

Where the ΔM_u is the mass loss of metal in uninhibited acid and ΔM_i is mass loss of metal in inhibited solution. The corrosion rate (mm year⁻¹) in can be obtained by the following equation [39].

$$\text{Corrosion rate (mm/year)} = \frac{\text{Mass loss} \times 87.6}{\text{Area} \times \text{time} \times \text{density}}$$

Area X time X density

Where mass loss in mg, Area is in cm² of metal surface exposed, the time is expressed in hours and metal density is expressed in gram/cm³ and 87.6 is the conversion factor. The activation energy obtained by the integrated form of Arrhenius equation [40, 41].

$$E_a = \frac{2.303 \times R \times (T_1 \times T_2)}{T_2 - T_1} \times \frac{\log C_2}{C_1}$$

Where C₂ and C₁ are corrosion rates at temperature T₂ and T₁ respectively. T is the absolute temperature and R is a gas constant. Free energy of adsorption was calculated using the following equation [42-44].

$$G = -RT \ln (55.5 K)$$

$$K = [\theta / C (1 - \theta)]$$

θ is degree of surface coverage on the surface of metal, C is the concentration of inhibitor, K is equilibrium constant, R is gas constant and T is temperature. The value of enthalpy of activation and entropy of activation can be determine according to the following equation[45]

$$CR = \left(\frac{RT}{Nh}\right) \exp\left(\frac{\Delta S^\circ}{R}\right) \exp\left(\frac{-\Delta H^\circ}{RT}\right)$$

Where h is the Planck's constant, N is the Avogadro's number, T is the absolute temperature and R is the universal gas constant. A plot of Log (CR/T) as a function of 1/T was made and straight lines were obtained. ΔH and ΔS were calculated from the slope and the intercept respectively from the linear plot.

Effect of temperature-Temperature affects the various reaction rates and, therefore, the effect on corrosion rates can be evaluated by conducting the experiment at different temperatures. Different ranges of temperature can be maintained with the help

of a constant temperature bath controlled automatically to the range of $\pm 1^\circ\text{C}$. Care should be taken to compensate the evaporation loss of electrolyte in order to measure the corrosion rate precisely

Table 1: Corrosion Rate (mmpy) and Inhibition Efficiency (η %) for extracts of *Acacia senegal* for mild steel in 2 N HCl from mass loss data at different temperatures and 24 hours immersion period.

| Inhibitor concentration | 303 K | | 313 K | | 323 K | |
|-------------------------|--------------------------------|----------------|--------------------------------|----------------|--------------------------------|----------------|
| | Inhibition efficiency η % | Corrosion Rate | Inhibition efficiency η % | Corrosion rate | Inhibition efficiency η % | Corrosion Rate |
| Uninhibited | - | 15.60 | - | 24.07 | - | 38.34 |
| Leaf | | | | | | |
| 0.12 | 60.80 | 6.12 | 53.41 | 11.22 | 43.90 | 21.51 |
| 0.24 | 65.05 | 5.45 | 56.32 | 10.52 | 50.65 | 18.92 |
| 0.36 | 72.21 | 4.34 | 60.87 | 9.42 | 54.38 | 17.49 |
| 0.48 | 76.09 | 3.73 | 68.47 | 7.59 | 59.73 | 15.44 |
| 0.60 | 83.50 | 2.57 | 73.37 | 6.41 | 63.92 | 13.83 |
| Gum | | | | | | |
| 0.12 | 60.92 | 6.10 | 58.88 | 9.90 | 43.44 | 21.68 |
| 0.24 | 66.63 | 5.21 | 62.36 | 9.06 | 48.32 | 19.81 |
| 0.36 | 69.30 | 4.79 | 66.55 | 8.05 | 52.28 | 18.30 |
| 0.48 | 73.06 | 4.20 | 68.54 | 7.57 | 61.82 | 14.64 |
| 0.60 | 77.55 | 3.50 | 73.22 | 6.45 | 66.95 | 12.67 |

Table 2: Kinetic – thermodynamic parameters for the corrosion of mild steel in 2N hydrochloric acid

| Inhibitor concentration | Activation energy Ea kJ/mol | | Free energy of adsorption $-\Delta G$ kJ/mol | | | Enthalpy change ΔH kJ/mol | Entropy change ΔS kJ/mol |
|-------------------------|-----------------------------|-----------|--|-------|-------|-----------------------------------|----------------------------------|
| | 303K-313K | 313K-323K | 303 K | 313K | 323K | | |
| Uninhibited | 34.20 | 39.12 | - | - | - | 0.0034 | -0.1678 |
| Leaf | | | | | | | |
| 0.12 | 47.82 | 54.74 | 16.57 | 16.33 | 15.83 | 0.2591 | -0.1587 |
| 0.24 | 51.78 | 49.38 | 15.28 | 14.83 | 14.70 | 0.2564 | -0.1580 |
| 0.36 | 61.19 | 52.02 | 15.10 | 14.26 | 14.01 | 0.2888 | -0.1556 |
| 0.48 | 56.01 | 59.69 | 14.89 | 14.38 | 13.82 | 0.2946 | -0.1540 |
| 0.60 | 71.95 | 64.65 | 15.49 | 14.42 | 13.70 | 0.3516 | -0.1500 |
| Gum | | | | | | | |
| 0.12 | 38.21 | 65.92 | 16.58 | 16.91 | 15.78 | 0.2615 | -0.1585 |
| 0.24 | 43.69 | 65.77 | 15.46 | 15.48 | 14.45 | 0.2716 | -0.1569 |
| 0.36 | 40.96 | 68.99 | 14.75 | 14.90 | 13.78 | 0.2772 | -0.1560 |
| 0.48 | 46.43 | 55.40 | 14.48 | 14.39 | 14.06 | 0.2573 | -0.1556 |
| 0.60 | 48.11 | 56.81 | 14.53 | 14.40 | 14.06 | 0.2655 | -0.1539 |

Table 3: Corrosion Rate (mmpy) and Inhibition Efficiency (η %) for extracts of *Acacia senegal* for mild steel in 2N H_2SO_4 from mass loss data at different temperatures and 24 hours immersion period

| Inhibitor concentration | 303 K | | 313 K | | 323 K | |
|-------------------------|--------------------------------|----------------|--------------------------------|----------------|--------------------------------|----------------|
| | Inhibition efficiency η % | Corrosion Rate | Inhibition efficiency η % | Corrosion Rate | Inhibition efficiency η % | Corrosion Rate |
| Uninhibited | - | 27.49 | - | 47.85 | - | 65.34 |
| Leaf | | | | | | |
| 0.12 | 52.59 | 13.03 | 49.54 | 24.14 | 39.10 | 41.45 |
| 0.24 | 63.75 | 9.97 | 59.9 | 19.19 | 47.21 | 35.93 |
| 0.36 | 69.72 | 8.32 | 62.3 | 18.04 | 52.27 | 32.48 |
| 0.48 | 78.75 | 5.84 | 70.71 | 14.02 | 57.02 | 29.25 |
| 0.60 | 86.59 | 3.69 | 79.01 | 10.04 | 68.01 | 21.77 |
| Gum | | | | | | |
| 0.12 | 58.03 | 11.54 | 55.03 | 21.52 | 41.21 | 40.01 |
| 0.24 | 67.07 | 9.05 | 64.06 | 17.20 | 53.05 | 31.95 |
| 0.36 | 73.04 | 7.41 | 69.04 | 14.81 | 57.57 | 28.88 |
| 0.48 | 79.02 | 5.77 | 75.03 | 11.95 | 66.22 | 22.99 |
| 0.60 | 88.09 | 3.27 | 83.05 | 8.11 | 70.73 | 19.92 |

Table 4: Kinetic – thermodynamic parameters for the corrosion of mild steel in 2N sulphuric acid

| Inhibitor concentration | Activation energy Ea kJ/mol | | Free energy of adsorption G kJ/mol | | | Enthalpy change H kJ/mol | Entropy change S kJ/mol |
|-------------------------|-----------------------------|-----------|------------------------------------|-------|-------|--------------------------|-------------------------|
| | 303K-313K | 313K-323K | 303 K | 313K | 323K | | |
| Uninhibited | 43.71 | 26.19 | - | - | - | 0.0033 | -0.1731 |
| Leaf | | | | | | | |
| 0.12 | 48.62 | 45.43 | 15.72 | 15.92 | 15.30 | 0.0045 | -0.1655 |
| 0.24 | 51.67 | 52.73 | 15.14 | 15.21 | 14.33 | 0.0050 | -0.1627 |
| 0.36 | 60.99 | 49.45 | 14.80 | 14.42 | 13.78 | 0.0054 | -0.1611 |
| 0.48 | 69.01 | 61.86 | 15.27 | 14.66 | 13.52 | 0.0064 | -0.1571 |
| 0.60 | 79.04 | 65.04 | 16.11 | 15.23 | 14.19 | 0.0071 | -0.1528 |
| Gum | | | | | | | |
| 0.12 | 49.15 | 52.15 | 16.28 | 16.50 | 15.54 | 0.0049 | -0.1641 |
| 0.24 | 50.60 | 52.08 | 15.51 | 15.67 | 14.96 | 0.0051 | -0.1620 |
| 0.36 | 54.62 | 56.11 | 15.20 | 15.20 | 14.36 | 0.0053 | -0.1600 |
| 0.48 | 57.44 | 55.02 | 15.31 | 15.23 | 14.57 | 0.0054 | -0.1579 |
| 0.60 | 71.54 | 75.55 | 16.45 | 15.92 | 14.54 | 0.0072 | -0.1513 |

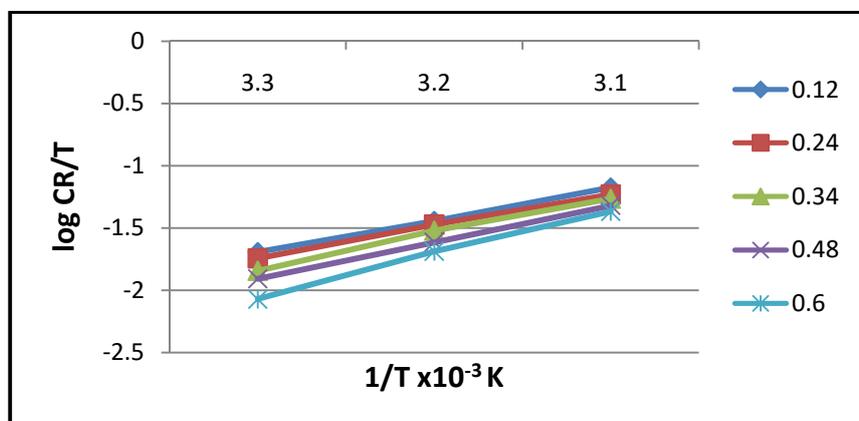


Figure 1: Transition state plot for mild steel corrosion in 2N HCl in the presence of different concentration of leaf extract

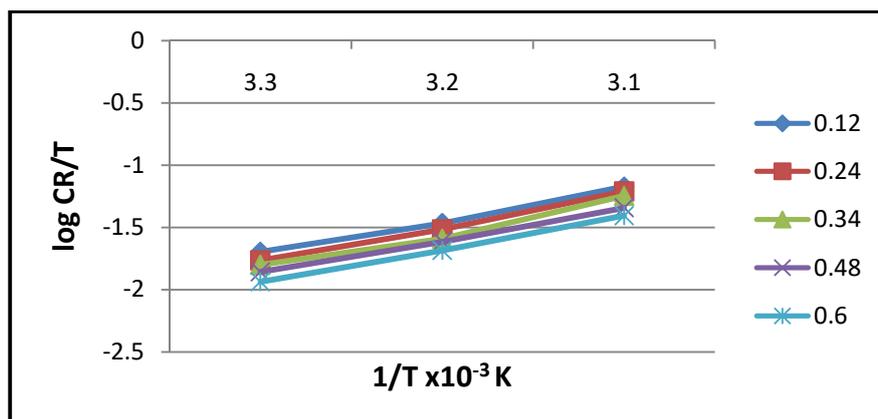


Figure 2: Transition state plot for mild steel corrosion in 2N HCl in the presence of different concentration of gum extract

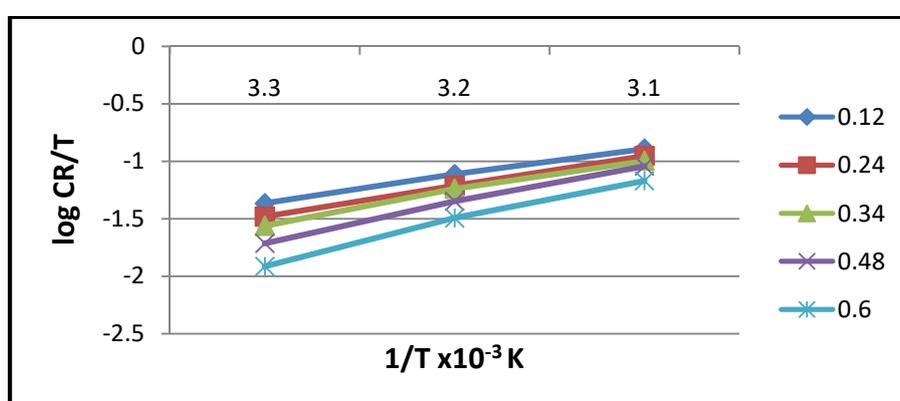


Figure 3: Transition state plot for aluminium corrosion in 2N H₂SO₄ in the presence of different concentration of leaf extract

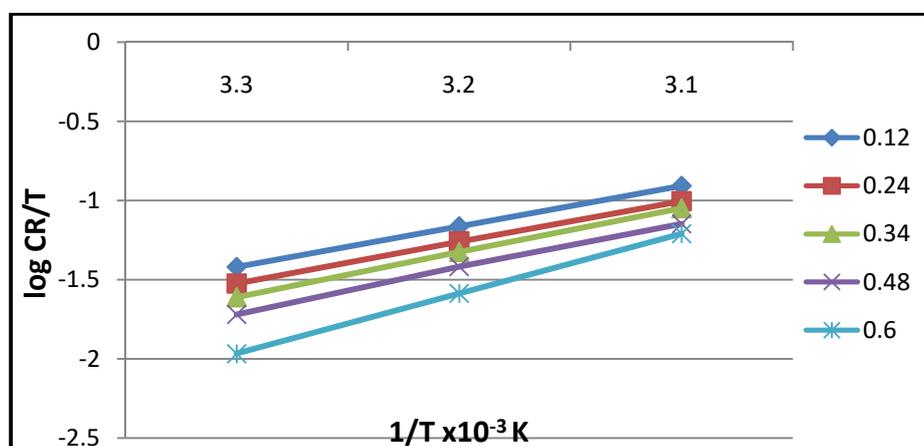


Figure 4: Transition state plot for aluminium corrosion in 2N H₂SO₄ in the presence of different concentration of gum extract

RESULT AND DISCUSSION

Corrosion rates and inhibition efficiency

All the inhibitors have reduced the corrosion rate to a significant extent. Corrosion rate and inhibition efficiency with or without inhibitor are shown in **Table 1 & 3**. The data

in the table shows corrosion rate of mild steel in H₂SO₄ and HCl reduces with an increase of inhibitor concentrations. The $\eta\%$ calculated from the mass loss measurement for 2N H₂SO₄ and HCl solution. It is found that the inhibition efficiency increases with

increase in inhibitor concentration for different plant extracts from 0.12% to 0.6% from the mass loss data. The inhibitors have shown the efficiency in the range from 52.59% to maximum 88.09% in 2N H₂SO₄ solution and from 60.80% to maximum 83.50% in 2N HCl respectively. The maximum efficiencies are obtained at high acid concentration (2N).

Mass loss at different temperatures

The mass loss data were obtained at different temperatures viz. 303K, 313K and 323K to investigate the effect of temperature and to determine whether there is change in the activation energy in presence and absence of the extracts in 2N of H₂SO₄ and HCl for mild steel. It is also observed that the corrosion rate increased to a large extent with rise in temperature. All the extracts provide inhibition even at 40° and 50 °C for mild steel in H₂SO₄ and HCl. However, the inhibition efficiency decreases from 2-10% for every 10°C rise of temperature.

The variation of inhibition efficiency, corrosion rate, enthalpy ΔH , entropy ΔS and free energy of adsorption ΔG activation energy in presence and absence of the extracts 2N H₂SO₄ and HCl for mild steel with temperature is shown in **Table 2 & 4**.

Inspection of tables reveals that the presence of acacia extracts increases the value of E_a as compare to the control (without extracts) indicating physical adsorption of the extracts

on the metal surface. The positive value of ΔH reflects that the endothermic nature of the metal dissolution process in the acid. The value of ΔS either in the presence or absence of the extract is negative, stating that the activation complex in the rate measuring step shows association rather than dissociation.

Adsorption and Thermodynamics studies

It has been reported that the adsorption of an organic compound on to the surface of a metal is dependent on the following physiochemical properties of inhibitor molecule, such as steric factors, functional groups, electron density at the donor atom and orbital character of donating electrons, nature of substrate metals and the type of interaction between organic molecules and the metallic surface as well [46]. In other words, the efficiency of an organic compound as corrosion inhibitor depends not only on the characteristics of the environment in which it works, metal surface and electrochemical potential at the interface, but also on the structure of the corrosion inhibitor itself; as the number of adsorption active centers, their charge density, molecular size, the mode of adsorption, the making of metallic complexes and the projected area of the inhibitor on the surface [47].

Adsorption isotherms are very useful in explaining the mechanism of organo-electrochemical reaction [48].

The value of free Gibbs adsorption energy of inhibitors, which is also presented on table can be determined using the following equation [51].

$$K = \frac{1}{55.5} \exp\left[\frac{-\Delta G^\circ}{RT}\right]$$

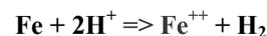
R= ideal gas constant, the value of 55.5 is the concentration of water in the solution in moles and T is the temperature in K.

Based on the values in table 2&4 it is shown that all of free Gibbs adsorption value, ΔG^0 , for each compound is negative, indicated the spontaneity of adsorption process on mild steel surface. The value of (ΔG_{ads}) until-20KJ/mol is consistently similar in case of electro-static interactions between the charged molecules and the charged metal surface (physical adsorption). Values which are more negative than -40KJ/mol, showing charge sharing/transfer from the inhibitor atoms to the surface of metal creating a coordinate type of bond (chemisorption). In the present study the low and negative value of ΔG_{ads} shown in table indicated the spontaneous adsorption of inhibitor on the surface of the mild steel. ΔG_{ads} values are less than -20KJ/mol, which show physical adsorption of inhibitors on the metal surface.

Mechanism of inhibition

Various metallic compounds are prone to acid corrosion. There is straight forward

mechanism of acid corrosion. In the case of mild steel, the Iron reacts with hydrogen ions to form ionic Iron and hydrogen gas. The reaction is show below:



The presence of corrosion inhibitor will have a profound effect on the corrosion rate. The addition of 0.1% to 0.2% of corrosion inhibitors can reduce the corrosion rate up to 95%. Generally, the organic inhibitors are absorbed on corroding metal surface through electron rich nitrogen atoms thus forming a chemisorbed monolayer of the inhibitor, which act as physical barrier between the metal and corrosive solution. The inhibitor may also form a chemical bond of the Lewis acid base type with the metal results in the formation of metal inhibitor complex on the metal surface which also retards the corrosion rate by acting as physical barrier. The protonated inhibitor molecules formed in the highly ionizing acids like the sulfuric acid and hydrochloric acid block the cathodic sites.

The OH⁻ group present in gum extract exerts a positive inductive effect, which increases the electron density at the nitrogen atom of protein moiety. This explains the higher inhibition efficiencies displayed by functional groups containing elements like Oxygen, Nitrogen, Sulphur and in some cases by Selenium and Phosphorus. The structure of the rest of the molecule can affect co-ordinate bond formation by its

influence on the electron density at the functional group. In the present study it is observed that total alkaloids of the natural products are adsorbed on the metal surface and decrease the surface area available for cathodic and anodic reactions to take place.

CONCLUSION

The study of extract of *Acacia senegal* has shown it to be effective inhibitors for the corrosion of mild steel in acid solution. Results from Mass loss method are showing that the inhibition efficiency is in correlation with increasing inhibitor concentration over the range of 0.12%-0.60%. It is concluded that-

- The plant extract of *Acacia senegal* is a good corrosion inhibitor for mild steel in acidic solution. This type of inhibitor is eco-friendly, biodegradable and less toxic therefore these type of inhibitors can be used to replace toxic chemicals
- The adsorption of *Acacia senegal* extract on mild steel surface in sulfuric acid obeys Langmuir adsorption isotherm which represent the monolayer formation of each compound on mild steel.
- Phenomenon of physical adsorption is proposed from the calculated values of kinetic/thermodynamic parameters.

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