# Corrosion Inhibition of Aluminium in Alkaline Medium using Vitex Negundo Leaves Extract

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## ABSTRACT

The influence of Vitex negundo leaves extract on the corrosion of aluminium in 1M NaOH solution was examined using chemical and electrochemical methods. It was found that the leaves extract inhibited corrosion of aluminium by forming an adsorption layer on aluminum surface. The inhibition efficiency of the extract increased with increase in its concentration and decreased with rise in temperature. Polarisation curves showed that all concentrations of the inhibitor studied were exhibiting mixed type action. Thermodynamic parameters such as adsorption equilibrium constant and free energy of adsorption were calculated from Langmuir adsorption isotherm and a physical adsorption mechanism was proposed. The associated energy of activation for corrosion, enthalpy of activation, and entropy of activation were calculated to explain metal inhibitor interaction.

**Keywords:** Corrosion inhibition, aluminium, weight loss, polarisation curves, AC impedance, adsorption.

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## INTRODUCTION

Aluminium is an important anode material for metal/air batteries, because of its high energy density. Due to the presence of a natural surface oxide film, aluminum exhibits very high corrosion resistance in various environments. But in alkaline solutions, the oxide film becomes non-protective and aluminium undergoes dissolution process [1-3].Organic and inorganic inhibitors are mostly used to control the dissolution of the protective oxide film in alkaline solutions.Organic compounds, containing mainly oxygen, nitrogen, and sulfur atoms, and having multiple bonds, are recognized as effective inhibitors for the corrosion of many metals and alloys [4-8]. However, these chemical inhibitors cause many pollution problems. Hence, the recent trend is to use environment friendly, cost effective and renewable natural inhibitors.Various parts of plants such as root, stem, bark, seed and leaves were reported as corrosion inhibitors of aluminium in several media [9-15]. The present study aims to investigate the corrosion inhibiting effect of Vitex negundo leaves extract for the corrosion of aluminium in alkaline medium using weight loss, hydrogen evolution, potentiodynamic polarisation and electrochemical impedance spectroscopy techniques. The work also aims to study the effect of temperature on the inhibitor efficiency of Vitex negundo leaves extract using weight loss experiments. Kinetic and thermodynamic studies were carried out to understand the mechanism of adsorption of inhibitor molecules on aluminium surface. *Vitex negundo* L. (Verbenaceae) is a hardy plant, flourishing mainly in the Indian subcontinent. All parts of the plant, from root to fruit, possess a multitude of phytochemical secondary metabolites which impart an unprecedented variety of medicinal uses to the plant.

## MATERIALS AND METHODS

Aluminium specimens of dimensions 5 X 1 X 0.1 cm were used for weight loss and gasometric techniques. The working electrode used for electrochemical polarisation and impedance measurements was cut in the form of a small cylindrical rod embedded in a Teflon gasket with an exposed area of 0.2826 $cm^2$. The aluminium rod is polished with different grades (320-1500) of emery papers and then washed with distilled water and dried with acetone. Each experiment was carried out with a newly polished electrode.

### Preparation of extract

The leaves of Vitex negunda were collected and dried in shade for 1 week. The dried leaves were ground well into powder. 10g of the dried sample was taken and refluxed in 100mL distilled water for 1 hour. The refluxed solution was filtered carefully and the filtrate was heated on a water bath to get the dried compound. The inhibitor solutions of 0.3, 0.6, 0.9, 1.2, and 1.5g/L concentrations were prepared from the dried compound using 1M NaOH solution. Double distilled water and Analar grade sample were used for the preparation of 1M NaOH solution [16].

### Weight loss test

Weight loss experiments were carried out as described earlier [17-20]. Aluminium specimens were immersed in 100mL of inhibited and unihibited solutions and allowed to stand for 1h at room temperature. Duplicate experiments were set up for each of the concentration. To study the effect of temperature on corrosion rate of aluminium the experiments were repeated at 40, 50 and $60^0$C and the results were compared with the results obtained at room temperature. The presence of a mixture of organic and resinous matter in the extract composition, such as glycosides,sterols and flavanoids, is attributed to the inhibitor action of the leaves extract. The corrosion rate and the inhibition efficiency were calculated using the following equations [19].

$$\text{Corrosion rate (mmpy)} = \frac{kW}{ATD} \qquad (1)$$

Where k = 8.76 $X10^4$(constant)

W= Weight loss in grams
A= Area in square cm
T= Time in hours and
D= Density in gm/cu.cm (2.70)

$$\text{Inhibition efficiency (\%)} = \frac{W_B - W_I}{W_B} \times 100 \qquad (2)$$

where, $W_B$ and $W_I$ are weight loss per unit time in the absence and presence of inhibitor.

### Gasometric technique

The dissolution of aluminium in 1M NaOH was monitored by hydrogen gas evolution in a gasometric assembly. The progress of the corrosion reaction of aluminium was recorded by carefully measuring the volume of hydrogen gas evolved at fixed time intervals. The inhibition efficiency was calculated from the hydrogen gas volume evolved during the immersion of aluminium specimen in 1M NaOH in the absence and presence of inhibitor [21,22].

### Polarisation and impedance measurements

Tafel polarisation method was adopted to record anodic and cathodic polarisation curves using an electrochemical workstation ( model CHI 608 CH Instruments USA) connected to a conventional three electrode cell. The scan rate was 1 mV/s and the measurements were made from -1700 to -1,300 mV.The inhibitor efficiency was calculated using the formula;

$$I\,E\,(\%) = \frac{i_{corr} - i^{*}_{corr}}{i_{corr}} x100 \qquad (3)$$

Where $I_{corr}$ and $I_{corr}^{*}$ are corrosion current densities in the absence and presence of Vitex negundo leaves extract.Impedance experiments were performed using the same electrochemical workstation mentioned above for polarisation studies. Impedance measurements were carried out at the open circuit potential in the frequency range of 100 kHz to 10 mHz. The charge transfer resistance ($R_{ct}$) and double layer capacitance values were calculated. The inhibitor efficiency was calculated using the formula [23]:

$$I.E\,(\%) = \frac{R^{*}_{ct} - R_{ct}}{R^{*}_{ct}} x100 \qquad (4)$$

where $R^{*}_{ct}$ and $R_{ct}$ are the charge transfer resistance in the presence and absence of Vitex negundo leaves extract.The electrochemical cell assembly used for both tests consisted of a saturated calomel electrode equipped with a Luggin capillary and a platinum foil as reference and counter electrodes respectively. Aluminium rod was used as the working electrode.

## RESULTS AND DISCUSSION

### Weight loss measurements

The weight loss results obtained for the corrosion of aluminium specimens for different concentrations of the extract in 1M NaOH solution at 30°C are given in Table1.It was observed from the table1 that increase in inhibitor concentration decreased the corrosion rate and therefore the corrosion inhibition strengthened. The increase in inhibitor efficiency may result from the fact that adsorption and surface coverage increases with the increase in concentration.As concentration increases more inhibitor molecules are adsorbed on the metal surface, thus resulting larger surface coverage. Hence the dissolution of aluminium decreases with increase in concentration of extract because the aluminium surface is efficiently separated from the alkaline medium[22].

Table1. Corrosion parameters for aluminium in 1M NaOH solution in the presence and absence of Vitex negundo leaves extract at 30°C.

| System / Concentrations ($gL^{-1}$) | Corrosion rate (mm/year) | Inhibition efficiency (%I) | Surface coverage (θ) |
|---|---|---|---|
| 1M NaOH | 212.21 | - | - |
| 0.3 | 93.34 | 56.0 | 0.56 |
| 0.6 | 83.23 | 60.7 | 0.60 |
| 0.9 | 71.80 | 66.1 | 0.66 |
| 1.2 | 63.29 | 70.1 | 0.70 |
| 1.5 | 46.27 | 78.1 | 0.78 |

**Gasometric technique**

The inhibitor action with increasing concentration is closely observed by the changes in $H_2$ gas evolution using gasometric technique. Fig-1 shows the variation of the volume of hydrogen gas evolved as a function of time when an aluminium specimen reacts with 1M NaOH in presence of different concentrations of Vitex negundo leaves extract. The volume of the hydrogen gas evolved is characterized by a sharp rise and approximately linear increase. The rate of hydrogen evolution is equivalent to the rate of aluminium dissolution and hence the inhibitor efficiency was calculated from the gasometry plot. The inhibition efficiency values increased with increasing concentrations of Vitex negundo leaves extract [21].

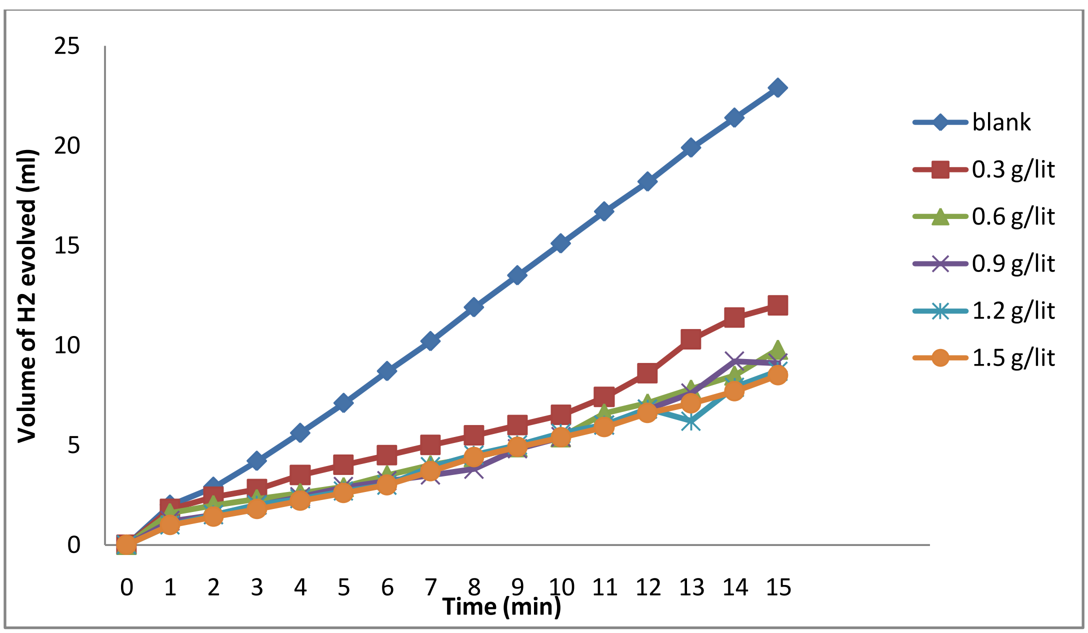


Fig 1: Plot of volume of hydrogen gas evolved against time for aluminium corrosion in the presence and absence of Vitex negundo leaves extract in 1M NaOH solution

Corrosion rate of aluminium determined by monitoring the $H_2$ gas evolution rate can provide information concerning the chemical nature of the surface film in situ at the metal corrodent interface.

**Potentiodynamic polarisation studies**

The anodic and cathodic polarisation curves for the corrosion of aluminum in the absence and presence of different concentrations of leaves extract are shown in figure2.Electrochemical parameters such as $E_{corr}$, $I_{corr,}$ anodic and cathodic Tafel slopes ($\beta_{a,}$ and $\beta_c$) are given in table2.

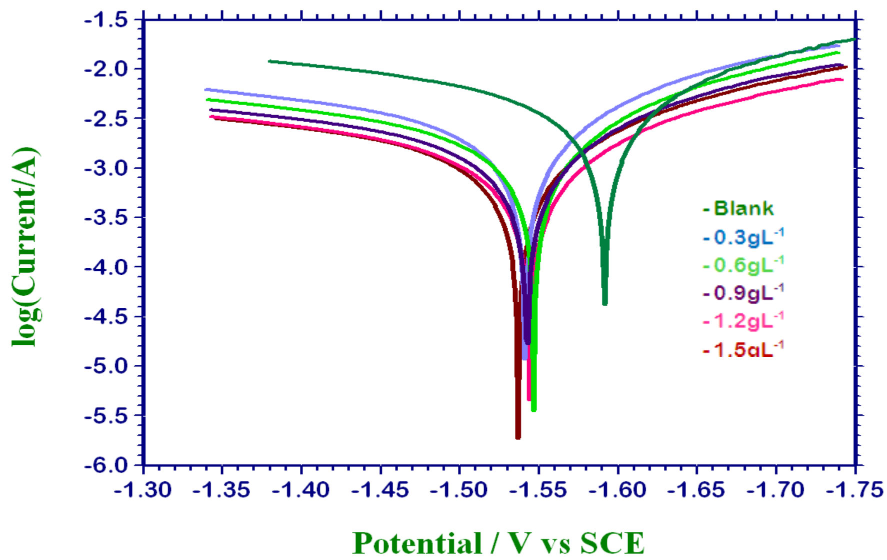


Fig 2: Polarisation curves for aluminium in 1MNaOH with and without Vitex negundo leaves extract

In the presence of different concentrations of inhibitor the corrosion current density was lowered significantly and this confirms the inhibitive nature of the leaves extract. $E_{corr}$ values were shifted to more positive side in the presence of inhibitor solutions indicating predominant anodic action of the inhibitor [5,23].

Table 2 Tafel polarisation parameters for the corrosion of aluminium in the presence and absence of inhibitor in 1M NaOH at room temperature.

| Concentration g /L | $-E_{corr}$ (mV) | $\beta_c$ ($mVdec^{-1}$) | $\beta_a$ ($mVdec^{-1}$) | $I_{corr}$ $mA.cm^{-2}$ | $R_{corr}$ mmpy | IE% |
|---|---|---|---|---|---|---|
| Blank | 158.8 | 5.421 | 185 | 313 | 209.01 | -- |
| 0.3 | 154.0 | 2.574 | 165 | 294 | 99.23 | 52.5 |
| 0.6 | 154.7 | 2.003 | 162 | 293 | 77.21 | 63.0 |
| 0.9 | 154.5 | 1.634 | 163 | 286 | 63.01 | 69.8 |
| 1.2 | 154.4 | 1.407 | 167 | 280 | 54.25 | 74.0 |
| 1.5 | 154.2 | 1.231 | 169 | 277 | 47.47 | 77.2 |

**AC Impedance studies**

The mechanism of inhibitor action of Vitex negundo leaves extract for the corrosion of aluminum in 1M NaOH was predicted by conducting impedance experiments. Figure 3 shows the Nyquist plots recorded in the absence and presence of different concentrations of leaves extract. The corresponding impedance results are given in Table 3. The increasing $R_f$ values and decrasing $C_{dl}$ values confirmed the inhibitive nature of the Vitex negundo leaves extract.

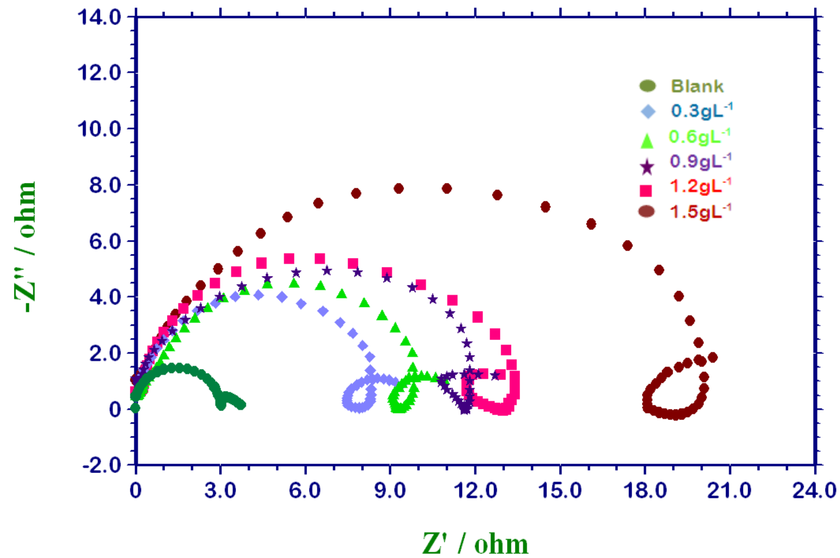


Fig 3: Nyquist plots for aluminium corrosion in 1MNaOH with and without Vitex negundo leaves extract

The semicircular nature of the Nyquist plot is due to the charge transfer process and the low frequency inductive loop is due to the growth and dissolution of the surface oxide film that controls the corrosion of aluminum. The capacitive and inductive loops showed in the Nyquist plots reveal the formation of metal inhibitor complex at metal/solution interface.The decrease in the thickness double layer charges was due to the formation of a complex at the metal surface [24].

Table3. Electrochemical impedance parameters and inhibition efficiencies for aluminium in 1 M NaOH solution in the absence and presence of Vitex negundo leaves extract

| Concentration of Vitex negundo extract ($gL^{-1}$) | $R_{ct}$ $\Omega\ cm^2$ | $C_{dl}$ ($\mu F/cm^2$) | Inhibition efficiency (%) |
|---|---|---|---|
| Blank | 3.09 | 88.76 | ---- |
| 0.3 | 8.04 | 15.54 | 54.0 |
| 0.6 | 9.26 | 11.39 | 60.2 |
| 0.9 | 11.23 | 10.53 | 67.2 |
| 1.2 | 12.13 | 9.18 | 69.6 |
| 1.5 | 18.19 | 8.00 | 79.7 |

The equivalent circuit model used to fit the experimental data is shown in figure 4. In the electric circuit $C_{dl}$ is double layer capacitance, $R_{ct}$ is the interfacial charge transfer resistance and $R_s$ is the solution resistance. The numerical values of $R_{ct}$ and $C_{dl}$ are determined by analysis of complex plane impedance plot and the equivalent circuit model by means of a computer program.

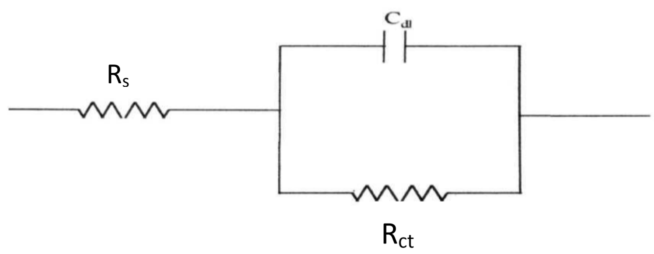


Fig 4: Equivalent electric circuit for a simple electrochemical cell.

**Kinetic and thermodynamic studies**

**Adsorption isotherm**

Inhibitors generally are adsorbed on the surface of the metal and thereby preventing access of the metal to the corrosive medium. The interaction between the inhibitor molecule and metal surface can be provided by means of adsorption isotherm. The inhibition efficiency is correlated to surface coverage and a 100% efficiency suggesting to full coverage(θ=1).The degree of surface coverage values for Vitex negundo leaves extract calculated from weight loss measurements (θ = % I/100), were used to determine its adsorption characteristics in 1M NaOH solution. To ascertain the nature of adsorption, the surface coverage values of Vitex negundo leaves extract obtained at 30°C-60°C were fitted into Langmuir adsorption isotherm. Langmuir adsorption isotherm is formulated as [5];

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C$$

Where, $\theta$ = degree of surface coverage

C= molar inhibitor concentration in the bulk solution.

$K_{ads}$ = Equilibrium constant of the adsorption process.

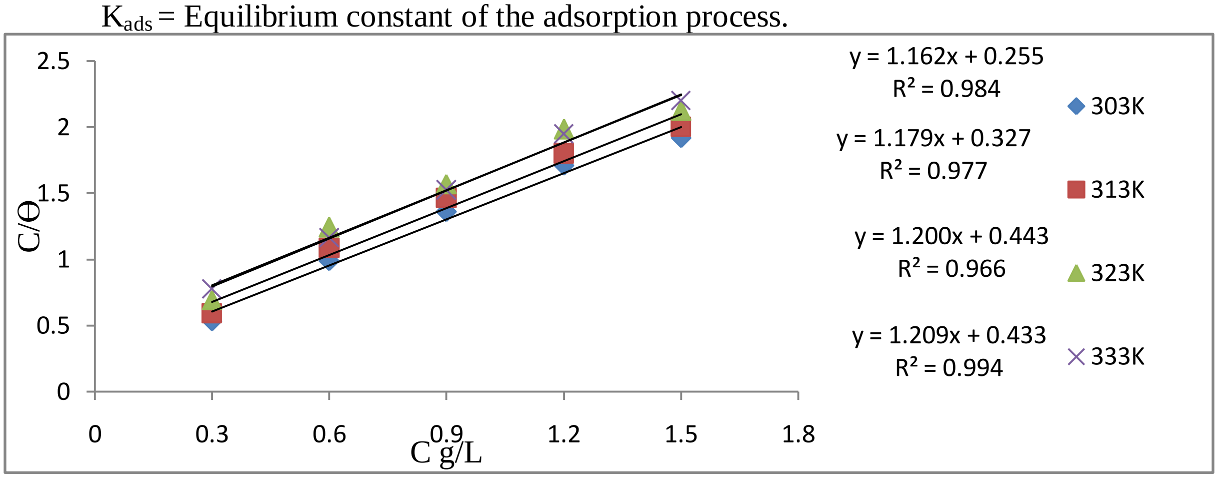


Fig: 5 Langmuir adsorption isotherm for Vitex negundo extract in 1M NaOH solution for aluminium at different temperatures.

The reciprocal of the intercept values obtained from the plot were used to calculate standard free energy adsorption $\Delta G_{ads}^0$. The adsorption equilibrium constant $K_{ads}$ is related to the standard free energy of adsorption, $\Delta G_{ads}^0$ with the following equation [7,25].

$$\Delta G_{ads}^0 = - RT \ln (55.5 \times K_{ads})$$

where 55.5 is the water concentration of the solution in $mol^{-1}$.

Table 4. Adsorption parameters of Vitex negundo leaves extract in 1M NaOH on aluminium at different temperature.

| Medium | $T^0C$ | $K_{ads}$ | $-\Delta G^0_{ads}$ KJ/mol |
|---|---|---|---|
| 1M NaOH | 30 | 3.90 | 13.35 |
| | 40 | 3.05 | 13.36 |
| | 50 | 2.25 | 12.96 |
| | 60 | 2.30 | 13.43 |

The values of $K_{ads}$ are low and decreasing with increase in temperature. This suggests the fact that adsorption strength decreases at higher temperatures and this is due to physical adsorption of inhibitor molecules on aluminium metal surface. The negative values of $\Delta G^0_{ads}$ obtained indicate the spontaneity of the adsorption process.

**Effect of temperature**

The effect of temperature on inhibition efficiency of the inhibitor was determined for 1M NaOH containing 0.3, 0.6, 0.9, 1.2, and 1.5g/L of the leaves extract at different temperatures ranging from 30°C to 60°C. The inhibition efficiencies of all concentrations of the additive were found to decrease with increasing temperature (30°C to 60°C).Inhibition efficiency (% I) decreased with increasing temperature as a result of fast dissolution of aluminium at higher temperature. This may be attributed to the fact that a higher temperature could lessen the inhibitor performance to a greater extent and this supports physical adsorption mechanism of Vitex negundo leaves extract on aluminium surface. Temperature influences the structure and thickness of the adsorbed film on metal [18].

Table 5. Effect of temperature on inhibition efficiency of Vitex negundo leaves extract in 1M NaOH solution for aluminium corrosion.

| Concentration of extract ($gL^{-1}$) | Inhibition Efficiency (%) | | | |
|---|---|---|---|---|
| | 30°C | 40°C | 50°C | 60°C |
| 0.3 | 56.0 | 50.7 | 43.1 | 38.6 |
| 0.6 | 60.7 | 55.2 | 48.2 | 51.5 |
| 0.9 | 66.1 | 61.4 | 57.3 | 59.0 |
| 1.2 | 70.1 | 66.6 | 60.4 | 61.6 |
| 1.5 | 78.1 | 74.8 | 70.5 | 68.2 |

Activation energy, $E_a$ is calculated with the help of the Arrhenius equation.

$$K = Ae^{-E_a/RT}$$

Where, k =corrosion rate
A= Arrhenius frequency factor.
$E_a$ = Activation energy for corrosion process.
R = Gas constant (8.314J/K mole)
T =Temperature in Kelvin

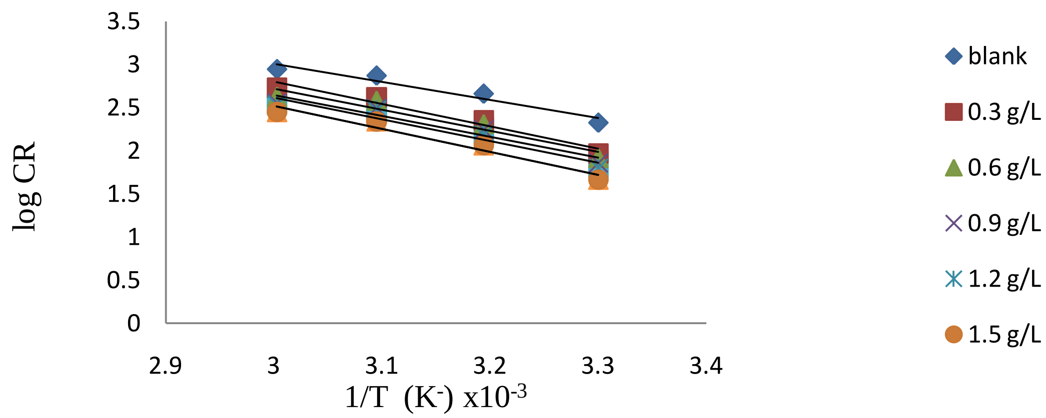


Fig 6: Arrhenius plot of log CR vs 1/T for aluminium corrosion in 1M NaOH in the absence and presence of Vitex negundo leaves extract

Plotting log CR against 1/T gave straight lines.The Ea values calculated from the slopes are given in the table 6. It is evident that for the corrosion of aluminium in 1M NaOH the $E_a$ value is 40.25kJ/mol and for the different concentrations of the inhibitors the Ea values are higher ranging from 49.88 to 51.09kJ/mol. Enthalpy of activation (ΔH*) and entropy of activation (ΔS*) are calculated from the slope and intercept of the line obtained from log k/T versus 1/T [12]. The values of Ea, ΔH* and ΔS* for aluminium corrosion in 1M NaOH in the presence and absence of different concentrations of Vitex negundo leaves extract are given in Table 8.

Table 6.Activation parameters obtained for various concentrations of Vitex negundo leaves extract.

| Concentration of extract ($gL^{-1}$) | $E_a$($kJ\ mol^{-1}$) | $\Delta H^o$($kJ\ mol^{-1}$) | $\Delta S^o$($JK^{-1}mol^{-1}$) |
|---|---|---|---|
| Blank | 40.25 | 37.61 | -75.36 |
| 0.3 | 49.88 | 47.23 | -50.44 |
| 0.6 | 46.89 | 44.25 | -60.96 |
| 0.9 | 46.02 | 43.38 | -65.06 |
| 1.2 | 48.11 | 45.47 | -59.33 |
| 1.5 | 51.09 | 48.45 | -52.20 |

The results obtained revealed that $E_a$ and $\Delta H^*$values increase in presence of Vitex negundo leaves extract, indicating a higher degree of surface coverage and higher protection efficiency attained due to raising the energy barrier for the aluminium corrosion reaction. Furthermore, the entropy of activation in the presence and absence of the inhibitor is small and negative. This may be attributed to the fact that the adsorption process is rather slow and activated complex in the rate determining step represents association rather than dissociation step, which means that a decrease in disordering occurs on going from reactants to activated complex.

## CONCLUSIONS

1. Vitex negundo leaves extract shows good inhibitive effect on aluminium corrosion in alkaline environment
2. Inhibition efficiency increases with the increase of extract concentration and decreases with rise in temperature.
3. Polarisation studies indicated that Vitex negundo leaves extract is showing predominant anodic action.
4. The values of standard free energy of adsorption suggest that the adsorption of inhibitor on aluminium surface occurred by physisorption mechanism.

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