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Ocimum sanctum (Tulsi) Leaves Extract as Corrosion Inhibitor for Aluminium in Hydrochloric acid Medium

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ABSTRACT: The inhibition of the corrosion of aluminium in hydrochloric acid solution by the leaves extract of *Ocimum sanctum* has been studied using weight loss, Potentiodynamic Polarization, Electrochemical Impedance Spectroscopic (EIS) techniques. Corrosion rate increases with the increase in acid concentration and temperature. As inhibitor concentration increases corrosion rate decreases while percentage of inhibition efficiency (I.E.) increases. The value of free energy of adsorption (ΔG^0_{ads}), heat of adsorption (Q_{ads}), energy of activation (E_a), enthalpy of adsorption (ΔF^0_{ads}) and entropy of adsorption (ΔS^0_{ads}) were calculated. The inhibition effect is discussed in view of *Ocimum sanctum* molecules adsorbed on the metal surface and it obeys Langmuir adsorption isotherm. Polarization curve indicates that inhibitor act as mixed type and the I.E. was found up to 85.17%. The results obtained showed that the leaves extract could serve as an effective green inhibitor for corrosion of aluminium in hydrochloric acid.

KEYWORDS: Aluminium, HCl, Ocimum sanctum, Corrosion inhibition, Weight loss, Polarization, EIS.

I. INTRODUCTION

Aluminium is one of the metals which used in different human activities and many of important applications, where it is the second most abundant metal after iron, it has a low atomic mass and negative value of standard electron potential. Aluminium is used in electronics due to it is super purity [1]. Although Al has an adhesive protective passivating oxide film, but this film has an atmospheric susceptibility, and consequently the metal dissolves readily in acidic and basic solutions concentrated above and below pH 4-9 [2,3]. 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 [4]. The importance of the study lies in the fact that natural plant products are non-polluting, ecofriendly, economic, less toxic and easily available than synthetic organic compounds. Ocimum sanctum (Tulsi) is a sacred plant and has various medicinal properties. There has been a growing trend in the use of natural products as corrosion inhibitor as they are environmentally safe, less toxic and readily available such as extracts of plant leaves for metals in acid cleaning process. Ocimum sanctum is a very common plant in Indian system, which has been used as anti-material, anti-bacterial and air purifier from ancient times in Indian homes. Its stem and leaves powder are used as medicine in many diseases like useful in blood glucose management, maintain a healthy digestive system, encourages efficient use of oxygen, enhances the efficacy of many therapeutic treatments etc. The chemical composition of Ocimum sanctum is highly complex, containing many vitamins A and C, calcium, zinc and iron, chlorophyll and many other phyto-nutrients are present in extract of Ocimum sanctum. Many workers [5-7] have studied the leaves extract of Ocimum sanctum as corrosion inhibitor. The aim of the present study is to investigate the corrosion inhibition effect of Ocimum sanctum for aluminium in various concentration of HCl medium by weight loss, effect of temperature, polarization and EIS techniques.



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II. EXPERIMENTAL

2.1. Preparation of sample and solution

The aluminium specimens with a chemical composition of 99.54 % Al, 0.090 % Si, 0.320 % Fe, 0.0012 % Cu, 0.0034 % Mn, 0.0014 % Mg, 0.0042 % Cr, 0.0046 % Ni, 0.0020 % Zn, 0.0079 % Ti, 0.0005 % Pb, and 0.0026 % Sn were used in the present study. The metal sheet, test specimens of size $5.0 \times 2.50 \times 0.198$ cm having an effective area of 0.279 dm²were used. The specimens were cleaned by washing with distilled water, degreased by acetone, washed once more with doubled distilled water and finally dried and weighted by using electronic balance. Hydrochloric acid was used as corrosive solution having concentration of 0.75, 1.0 and 1.25 M prepared by diluting analytical grade of HCl purchased from Merck using double distilled water.

2.2. Preparation of extract

The leaves of *Ocimum sanctum* were cut into small pieces, dried in a hot air oven at 80°C for 2 h and ground into powder. From this 10 g sample was refluxed in 100 mL distilled water for 1 h, filtered carefully and made to 100 mL using double distilled water. The concentration of the stock solution is expressed in terms of g/L [7].

2.3. Weight loss measurements

For weight-loss measurement, the aluminium specimen having an area of 0.2797 dm² were each completely suspended in 230 ml of 0.75, 1.0 and 1.25 M HCl solution with and without different *Ocimum sanctum* leaves extract concentrations using glass hooks at 301 ± 1 K for 24h. The coupons were retrieved after 24h, washed with distilled water, dried well and reweighed. From the weight loss data, corrosion rate (CR) was calculated.

2.4. Temperature effect

To study the effect of temperature on corrosion rate, aluminium coupons were completely immersed in 230 ml of 0.75M HCl solution without and with different concentrations of *Ocimum sanctum* leaves extract at 313, 323 and 333K for 2h. From the data corrosion rate, inhibition efficiency (I.E.), activation energy (E_a) and heat of adsorption (Q_{ads}) and free energy of adsorption (ΔG_{ads}^0) were calculated.

2.5. Potentiodynamic polarization measurements

Both the potentiodynamic and EIS measurement were carried out using CHI608C-series, U.S. Model with CHinstrument. For polarization study, metal specimens were immersed with and without *Ocimum sanctum* leaves extract in 0.75 M HCl solution. In the electrochemical cell aluminium specimens having an area of 1 cm² was used as a working electrode, Ag/AgCl electrode as a reference electrode and platinum electrode as an auxiliary electrode. Before each electrochemical measurement, the working electrode were allowed to stand for 65 min. in test solution to establish a steady-state open circuit potential (OCP). The polarization curves were plotted with current Vs potential. An anodic and cathodic polarization curve gives corresponding anodic and cathodic Tafel lines. The intersect point of Tafel lines gives the corrosion potential (E_{corr}) and corrosion current (i_{corr}) [8]. Cathodic Tafel slope (β c) and anodic Tafel slope (β a) were calculated from the software installed in the instrument.

2.6. Electrochemical Impedance Spectroscopy (EIS) measurements

EIS measurements were made at corrosion potentials over a frequency range of 1 to 10^5 Hz by a sine wave with potential perturbation amplitude of 5 mV. A graph was drawn by plotting real impedance (Z') versus imaginary impedance (-Z'').From the Nyquest plots of Z' Vs-Z'' the charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) were calculated. An experiment was carried out in absence and presence of inhibitor.

III.RESULTS AND DISCUSSION

3.1. Weight loss experiments

The weight loss experiment was carried out in 0.75, 1.0 and 1.25 M HCl solution containing 0.6, 0.8, 1.0 and 1.2 g/L concentration of *Ocimum sanctum* leaves extract at 301 ± 1 K for a exposure period of 24h was investigated. Corrosion rate (C.R.) was calculated using following equation:



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| C.R. (mg/dm^2d) = Weight loss (gm) x 1000 / Area in dm ² x day | (1) |
|---|-----------|
| Inhibition efficiency (I.E.) was calculated by using following equation: | |
| I.E. (%) = $\left\{\frac{(w_u - w_i)}{w_u}\right\} \times 100$ | (2) |
| Where, W_u = Weight loss in absence of inhibitor, W_i = Weight loss in presence of i | nhibitor. |
| The degree of surface coverage (θ) of the aluminium specimen for different conce | ntration |

The degree of surface coverage (θ) of the aluminium specimen for different concentration of HCl solution have been evaluated by weight loss experiments using the following equation:

$$\theta = \frac{(W_u - W_i)}{W_i}$$

(3)

Results showed in Table-1 indicates that as the acid concentration increases corrosion rate increase while I.E.decreases. Corrosion rate was 2195.20, 6163.74 and 8283.87 mg/dm²d corresponding to 0.75, 1.0 and 1.25 M HCl concentrations respectively for an immersion period of 24 h at 301 ± 1 K (Figure-1). At constant acid concentration, as the inhibitor concentration increases corrosion rate decreases while I.E. increases. For example, in 0.75 M HCl solution, the I.E. was found to be 70.19, 73.45, 78.01 and 85.17 % corresponding to 0.6, 0.8, 1.0 and 1.2 g/L inhibitor concentration respectively (Table-1 and Figure-2).

Table1 :Effect of HCl concentration on corrosion rate (CR) and inhibition efficiency (IE) of aluminium having different concentration of *Ocimum sanctum* leaves extract.

| Inhibitior | | | Acid conc | entration | | |
|------------------|------------------------------|-------------|------------------------------|-------------|------------------------------|-------------|
| concen- | 0.75 M | | 1.0 | Μ | 1.25 M | |
| tration (g/L) | CR (mg/dm ² d) | I.E. (%) | CR (mg/dm ² d) | I.E. (%) | CR (mg/dm ² d) | I.E. (%) |
| Blank | 2195.20 | - | 6163.74 | - | 8283.87 | - |
| 0.6 | 654.27 | 70.19 | 2123.70 | 65.54 | 4654.98 | 43.80 |
| 0.8 | 582.76 | 73.45 | 1734.00 | 71.86 | 3775.47 | 54.42 |
| 1.0 | 482.65 | 78.01 | 1365.74 | 77.84 | 3049.69 | 63.18 |
| 1.2 | 325.34 | 85.17 | 1076.15 | 82.54 | 1669.64 | 79.84 |

Table2 :Inhibition efficiency (I.E.), Corrosion rate(p) and Surface coverage(θ) of*Ocimum sanctum* leaves extract on aluminium in 0.75 M HCl for
an immersion period of 24h at 301 ± 1 K.

| Inhibitor | Inhibitor Concentration (g/L) | C.R. (ρ) (mg/dm ² d) | log p | I.E. (%) | Surface coverage (θ) | C/θ |
|------------------------------|-------------------------------------|------------------------------------|-------|-------------|----------------------------|------|
| Blank | - | 2195.20 | 3.34 | - | - | - |
| Ocimum sanctum (Tulsi) | 0.6 | 654.27 | 2.81 | 70.19 | 0.70 | 0.85 |
| | 0.8 | 582.76 | 2.76 | 73.45 | 0.73 | 1.09 |
| | 1.0 | 482.65 | 2.68 | 78.01 | 0.78 | 1.28 |
| | 1.2 | 325.34 | 2.51 | 85.17 | 0.85 | 1.41 |



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Fig.1 :Corrosion rate of aluminium in 0.75 M HCl solution in absence and presence of different concentration *Ocimum sanctum* leaves extract for 24 h.





3.2. Temperature effect

To investigate the influence of temperature on corrosion of aluminium, the weight loss experiments were also carried out at 313, 323 and 333K in 0.75 M HCl in absence and presence of *Ocimum sanctum* for an immersion period of 2h. The results in Table-3 shows that corrosion rate increases with rise in temperature. Corrosion rate was 21451.44, 26642.76 and 43417.92 mg/dm²d corresponding to 313, 323 and 333K respectively. The value of energy of activation (E_a) has been calculated with the help of Arrhenius equation [9].

$$\log \frac{\rho_2}{\rho_1} = \frac{E_a}{2.303R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right)$$
(4)

Where, ρ_1 and ρ_2 are the corrosion rate at temperature T_1 and T_2 respectively.



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Table3 :Temperature effect on corrosion rate (CR), activation energy (E_a) and heat of adsorption (Q_{ads}) for aluminium in 0.75 M HCl in absence and presence of *Ocimum sanctum* leaves extract for an immersion period of 2 h.

| | | Temperature | | | | | | Q _{ads} | |
|---------|----------------------|-------------|----------------------|-------|----------------------|-------|-------------------------------|------------------------|---------|
| concent | 313 K | | 323 K | | 333 K | | (E _a) From | (kJmol ⁻¹) | |
| -ration | CR | I. E. | CR | I. E. | CR | I. E. | Equation | 313-323 | 323-333 |
| (g/L) | mg/dm ² d | (%) | mg/dm ² d | (%) | mg/dm ² d | (%) | (4) (kJmol ⁻¹) | K K | К |
| Blank | 21451.44 | - | 26642.76 | - | 43417.92 | - | 30.92 | - | - |
| 0.6 | 3904.08 | 81.80 | 6821.52 | 74.39 | 12141.48 | 72.03 | 49.22 | -36.68 | -10.77 |
| 0.8 | 3046.08 | 85.80 | 5620.20 | 78.90 | 10253.76 | 76.38 | 52.62 | -40.34 | -12.99 |
| 1.0 | 2531.28 | 88.20 | 5062.56 | 80.99 | 8494.80 | 80.43 | 52.26 | -47.25 | -03.21 |
| 1.2 | 1716.12 | 92.00 | 4032.84 | 84.86 | 7336.32 | 83.10 | 62.65 | -60.41 | -11.69 |

Results given in Table-3, indicates that the values of E_a were higher in inhibited acid ranging from 49.22 to 62.65 $kJmol^{-1}$ than E_a value for uninhibited acid (30.92 $kJmol^{-1}$) which indicates physical adsorption of the inhibitor on metal surface and the adsorption of inhibitor causes an increase in the E_a value of the process [10]. Results of Table-3 indicates that as temperature increases, rate of corrosion increase while percentage of I.E. decreases. The value of E_a were also calculated from the slope of the Arrhenius plot of log ρ versus 1/T x 1000 (Figure-3) shows good agreement with the calculated values.



Fig.3 :Arrhenius plots for aluminium in 0.75 M HCl in absence and presence of the different concentration of *Ocimum sanctum* leaves extract.

The values of heat of adsorption (Q_{ads}) were calculated by using the following equation [11].

 $Q_{ads} = 2.303R \left[\log \left(\frac{\theta_2}{1 - \theta_2} \right) - \log \left(\frac{\theta_1}{1 - \theta_1} \right) \right] \left[\frac{T_1 T_2}{T_2 - T_1} \right]$ (5) Where θ_1 and θ_2 are the fraction of the metal surface covered by the inhibitor at temper

Where, θ_1 and θ_2 are the fraction of the metal surface covered by the inhibitor at temperature T_1 and T_2 respectively. It is evident that in all cases, the value of Q_{ads} were negative and ranging from -03.21 to -60.41 kJmol⁻¹. The negative values shows that the adsorption and hence the I.E. decreases with rise in temperature.



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3.3. Adsorption isotherms

The application of adsorption isotherms allow to analyse the interaction mode in the inhibition process between adsorbent and adsorbed [12]. The Langmuir isotherm assumed that the adsorbed molecules occupy only one site and there was no interaction with other adsorbed molecules in metal surface [13].

The surface coverage ' θ ' value was calculated by using equation- 3. A plot of inhibitor concentration C_{inh} versus C_{inh}/θ was presented in Figure-3 which gives straight line with slope values equal to unity indicates that the system follows Langmuir adsorption isotherm [14]. This isotherm can be represented as,

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + 0$$

(6)

Where, K_{ads} is the equilibrium constant and C_{inh} is the inhibitor concentration.



Fig.4 :Langmuir adsorption isotherm plot for aluminium in 0.75 M HCl containing Ocimum sanctum leaves as green inhibitor at 301 K.

Free energy of adsorption (ΔG^{o}_{ads}) was determined by the Langmuir isotherm was given by a plot of C/ θ versus C (Figure-4) [15]. From the intercepts of the straight line on the C/ θ axis, K_{ads} can be calculated which was related to ΔG^{o}_{ads} as given by the following equation[16,17].

$$\Delta G^{o}_{ads} = -RT \ln (55.5 K_{ads})$$

(7)

Where , R is the gas constant, T is the concentration of water in solution in Molar [18], K_{ads} is the equilibrium constant of the adsorption/desorption process. The mean ΔG^{o}_{ads} value was found negative (-15.27 kJmol⁻¹) suggest that the adsorption mechanism of *Ocimum sanctum* on aluminium in 0.75 M HCl at the studied temperatures is physisorption with adsorptive layer having electrostatic character [19]. This is concluded on the fact that the values of ΔG^{o}_{ads} -20 kJmol⁻¹ are consistant with physisorption, while those around -40 kJmol⁻¹ or higher are associated with chemisorption [20].

The enthalpy of adsorption (ΔH^{o}_{a}) and entropy of adsorption (ΔS^{o}_{a}) were calculated using the equations (8) and (9), $\Delta H^{o}_{a} = E_{a} - RT$ (8)

$$\Delta S_{a}^{\circ} = \Delta H_{a}^{\circ} - \Delta G_{a}^{\circ} / T$$
⁽⁶⁾
⁽⁶⁾
⁽⁶⁾
⁽⁷⁾
⁽⁶⁾
⁽⁷⁾
⁽⁶⁾
⁽⁷⁾

Results indicates that values of ΔH^{o}_{a} were positive and increase in presence of inhibitor indicating a higher degree of surface coverage and higher protection efficiency attained due to raising the energy barrier for the aluminium corrosion reaction. The enthalpy change ΔH^{o}_{a} was positive and ranging between 46.24 to 71.78 kJmol⁻¹indicating the endothermic nature of the reaction suggests that higher temperature favours the corrosion process. Positive values of ΔS^{o}_{a} ranging from 0.190 to 0.277 kJmol⁻¹indicates that corrosion process is entropically favourable.



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3.4. Potentiodynamic polarization study

Figure-5 represents the potentiodynamic polarization curves of aluminium in 0.75 M HCl in absence and presence of *Ocimum sanctum* leaves extract. Electrochemical parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}), anodic Tafel slope (βa), cathodic Tafel slope (βc) and percentage inhibition efficiency (I.E.) were given in Table-4.

Table4 :Potentiodynamic polarization data and Inhibition efficiency (I.E.) of *Ocimum sanctum* leavesextract as green inhibitor for aluminium in 0.75 M HCl.

| System | Б | T | | Tafel slope (V / decade) | IE(% Calculated |) from | |
|---------------------------|--------------------------|----------------------------------|-----------------|-----------------------------|--------------------|------------------------|--------------------------|
| | E _{corr} (V) | I_{corr} ($\mu A / cm^2$) | Anodic (+βa) | Cathodic (-βc) | β (V) | Polarization method | Weight loss method |
| Blank | -0.895 | 9156 | 6.706 | 5.621 | 1.329 | - | - |
| Ocimum sanctum (Tulsi) | -0.875 | 2156 | 8.491 | 5.400 | 1.435 | 76.45 | 85.17 |





From Table-4, it was observed that the addition of *Ocimum sanctum* leaves extract in acid solution indicates the significant decrease in corrosion current density (i_{corr}) and decrease in corrosion rate with respect to blank. There is significant change in the anodic and cathodic slopes after the addition of the inhibitor. In general, an inhibitor is anodic or a cathodic if the variation of E_{corr} against the blank is higher or above than 85 mV [21,22]. In this study, the displacement of the E_{corr} was about 20 mV (Table 4), these result suggest that the *Ocimum sanctum* should be considered as a mixed type inhibitor.

Inhibition efficiency (I.E.) from polarization study was calculated using following equation [23].

I.E. (%) =
$$\frac{i_{corr(uninh)} - i_{corr(inh)}}{i_{corr(uninh)}}$$

(10)



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3.5. Electrochemical impedance spectroscopy (EIS) measurements

EIS technique was studied to investigate the growth of the film formed on aluminium surface by *Ocimum sanctum* extract in 0.75 M HCl.

Nyquist plots for the corrosion of aluminium in 0.75MHCl solution in absence and presence of *Ocimum sanctum* leaves extract was examined by EIS method at room temperature was shown inFigure-6 and EIS parameters in Table-5.

Table5 :EIS parameters for corrosion of Aluminium in 0.75 M HCl containing Ocimum sanctum leaves extract.

| System | \mathbf{R}_{ct} | C_{dl} | I.E. (%) Calculated from | | |
|---------------------------|-------------------|--------------------|-----------------------------|--------------------|--|
| | (12 cm) | (µ r / cm) | EIS method | Weight loss method | |
| Blank | 84 | 45.130 | - | - | |
| Ocimum sanctum (Tulsi) | 250 | 4.899 | 89.14 | 85.17 | |



Fig.6 :Nyquist plot for aluminium (a) in 0.75 M HCl (Blank) (b) in 0.75 M HCl in presence of 1.2 g/L *Ocimum sanctum* leaves extract.

It is observed from Figure-6 that the impedance diagram is almost semi circular. The difference has been attributed to frequency dispersion. The semicircular nature of the plots indicates that the corrosion process was mainly charge transfer controlled [24].

The diameter of capacitive loop in the presence of inhibitor is bigger than that in the absence of inhibitor. The high frequency capacitive loop is related to the charge transfer resistance (R_{ct}). To calculate the double layer capacitance (C_{dl}), the frequency at which the imaginary component of the impedance is maximum was found as presented in the following equation [25].

$$C_{dl} = \frac{1}{2\pi F_{max}R_{ct}}$$

(11)

Where 'f' is the frequency at the maximum height of the semicircle on the imaginary axis and R_{ct} is the charge transfer resistance [26].

Inhibition efficiency (I.E.) from EIS method was calculated using following equation:

$$I.E.(\%) = \frac{Cdl(uninhi.) - Cdl(inhi.)}{Cdl(uninhi.)} x \ 100$$
(12)

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The addition of inhibitor increase R_{ct} value from 84 to 250 $~\Omega\ cm^2$, at the same time it decreases the C_{dl} values from 45.13 to 4.899 μF / cm^2 which is due to the adsorption of inhibitor on the metal surface. The results suggest that the inhibitor acts by the formation of a physical protective layer on the surface that retards the charge transfer process and therefore inhibit the corrosion reaction, leading to increase in R_{ct} values. Moreover, the adsorbed inhibitor species decrease the electrical capacity of electrical double layer values at the electrode/solution interface and therefore decrease the value of C_{dl} [27].

3.6. Mechanism of corrosion

Generally, aluminium dissolves in acid solutions due to hydrogen evolution type of attack, the reactions taking place at the microelectrode of the corrosion cell being represented as: [28,29]:

| $Al \rightarrow Al^{+3} + 3 e^{-}$ (anodic reaction) | (13) |
|--|----------|
| $H^+ + e^- \rightarrow H_a$ (cathodic reaction) | (14) |
| Followed by the reactions: | |
| $H + H \rightarrow H_{2(g)} \qquad \dots$ | (15) |
| | |

| The following secondary reactions can also take place in acid solutions [30]: | |
|---|------|
| $2M + 2H^+ \rightarrow H_2 + 2M^+ (anodic) \qquad \dots$ | (16) |
| $O_2 + 4H^+ + 4e^- \rightarrow 2H_2O$ (cathodic) | (17) |

3.6. Mechanism of inhibition

Major chemical constituents responsible for physicochemical action of *Ocimum sanctum* are volatile oil (0.1 to 0.9%), Eugenol (60–70%), Cavarcol (about 3.0%), Eugenol methyl ether (20%) and other minorchemical constituents are like alkaloids, glycoside, saponin, tannin, maleic acid, ursolic acid, citric acid and tartaric acid [6]. Structure of some of these compounds are shown below.





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These compounds contain oxygen and nitrogen atoms which possess lone pairs of electrons that may facilate the formation of dative bonds acting as centres for adsorption, thus, creating barrier between the metal suface and the corrosive media [31]. From Table-3, it can also be shown that the I.E. decreases with an increase in temperature. Such behaviour can be interpreted in terms of the fact that the phytochemical molecules are physically adsorbed on the surface of metal. Since, a physisorption mechanism is indicative of a weak adsorption bond, it may be possible that at higher temperatures, the decrease in I.E. may be a result of increased agitation of the solution resulting from higher rates of hydrogen evolution, thereby reducing the ability of the molecules to be adsorbed on the surface of the metal.

IV. CONCLUSION

As acid concentration increases corrosion rate increases while I.E. decreases. At constant acid concentration, as inhibitor concentration increases corrosion rate decreases while I.E. increases. As temperature increase corrosion rate increases while I.E. decreases. The leaves extract of *Ocimum sanctum* showed maximum I.E. of 85.17 % at an optimum concentration of 1.2 g/L. The values of E_a obtained in the presence of the extract were higher compared to the blank acid solution which indicates that inhibitor was more effective at lower temperature. The values of ΔG^o_{ads} were negative, which reveals the spontaneous adsorption of inhibitor cover both anodic and cathodic regions through general adsorption following Langmuir isotherm. Polarization curves indicates that the *Ocimum sanctum* leaves extract act as mixed type of inhibitor.

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