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# The effect of aegle marmelos leaves extract on corrosion inhibition of aluminium in alkaline solution

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## ABSTRACT

*The effect of aegle marmelos leaves (AML) extract on the corrosion inhibition of aluminium in 1N NaOH solution was studied using chemical and electrochemical techniques. It was found that the inhibition efficiency increased with the increase of AML extract upto 800 ppm. Beyond this concentration, corrosion inhibition efficiency was decreased from 87.6 to 84.7% It indicates that 800ppm is the optimum concentration to get maximum corrosion protection for aluminium 1N NaOH. The results obtained from chemical and electrochemical measurements are in reasonably good agreement. The potentiodynamic polarization studies reveal that the AML extract act as mixed type inhibitor. Adsorption of AML extract on aluminium surface follows Langmuir adsorption isotherm. The surface characteristics of the inhibited and uninhibited aluminium were investigated by scanning electron microscope studies.*

**Keywords:** Corrosion inhibitors, Aluminium corrosion, Aegle marmelos leaves extract, Langmuir adsorption isotherm, Mixed type inhibitors.

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## INTRODUCTION

The corrosion behavior of pure aluminium and its alloys in alkaline solutions have been extensively studied in the development of aluminium anodes for aluminium/air batteries [1-3]. Aluminium corrosion within the battery cause many problem; (i) passivates the cathode active material, (ii) its solid products increase the self-discharge and the electrical resistance,(iii) its soluble products contaminate the electrolyte and increase the self-discharge rate, and (iv) the dissolved $Al^{3+}$ ions migrate to the counter anode and reductively deposit [4]. Therefore, in most aqueous solutions, the anodic overvoltage for the dissolution reaction of aluminium is very high. In solution containing aggressive anions or in highly alkaline solutions, the dissolution procedure occurs in a much easier way. Although there is a high amount of dissolution, which is favorable, there arises a problem of high hydrogen evolution which in turn commercial application of the situation. Thus, commercial application of aluminium and its alloys requires minimizing the over voltage for the anodic process, while increasing it for the cathodic process [5]. Most of the Research works have been made by using chemical compounds as an inhibitor to study the corrosion properties of the metal. [6]. The use of chemical inhibitors has been limited because of the environmental threat. Recently, due to environmental regulations, plant extracts have again become important because they are environmentally acceptable, readily available and renewable source for a wide range of needed inhibitors. For instance, henna leaves [7] gossipium higgutum [8], gum arabic [9] phyllanthus amarus [10], ipomoea involucrate [11], hibiscus sabdoriffa leaves [12] have been studied as effective corrosion inhibitors for aluminium in alkaline medium. This paper reports the influence of AML extract on the corrosion of aluminium in 1N alkaline solution by weight loss, gasometric, potentiodynamic polarization and AC-impedance studies to find out the inhibition efficiency of the inhibitors and a suitable mechanism regarding the mode of inhibition was also proposed. Surface examination on the aluminium in the absence and presence of inhibitor was made to confirm the formation film on the surface of aluminium.

## MATERIALS AND METHODS

### Material preparation

Aluminium strips of 4.5% and 2cm × 0.2cm containing 1.5% Pb,0.1% Ga, 1% In and the remainder Al were used for weight loss, gasometric and hydrogen permeation studies. The strips were mechanically polished and degreased with acetone before use. A cyclindrical aluminium rod of the same composition embedded in a Teflon rod with an exposed area of $0.5cm^2$ was used for potentiodynamic polarization studies and AC impedance measurements. Analar grade NaOH and double distilled water were used to prepare the solutions.

### Preparation of leaves of adathoda vasica extract

The aegle marmelos leaves (AML) were collected and cut into small pieces and they were dried in an air oven at 80°C for 2 hrs. They were ground well into powder. From this, 10g of the sample was refluxed in 100mL distilled water for 1 hour. The refluxed solution was filtered carefully and the filtrates were heated on water bath to evaporate fully the moisture content to get the dired compound [13]. the inhibitor concentrations of 200, 400, 600, 800 and 1000ppm were prepared using 1N NaOH solution.

### Techniques used for the study

### Weight loss measurements

Weight loss measurements were carried out as described elsewhere [14]. Aluminium specimens were immersed in 100ml of inhibited and uninhibited solutions for 2 hours at 30°C. The corrosion rate (mmpy) and the inhibiton efficiency were calculated using the following equations;

$$\text{Corrosion rate (mmpy)} = KW/ATD$$

Where, $K = 8.76 \times 10^4$ (constant), W = weight loss in g, A= area in square cm, T= time in seconds and D= density in gm / cu.cm (2.70)

$$\text{Inhibition Efficiency (\%)} = W_B - W_I / W_B \text{ x100}$$

Where, $W_B$ and $W_I$ are weight loss per unit time in the absence and presence of AML extract.

Weight loss measurements were also performed at various immersion time from 2 hours to 24 hours in an optimum concentration of the AML extract at 30°C. From the initial and final weight of the specimen, the loss in weight was calculated and the efficiency of inhibitor at various immersion time was calculated.

### Determination of surface coverage

The degree of surface coverage (θ) was calculated from the weight loss measurement results using the formula [15];

$$\text{Surface coverage } (\theta) = \frac{W_B - W_I}{W_B}$$

Where, $W_B$ is the weight loss in the absence of the extract, $W_I$ is the weight loss in the presence of the extract. The data were tested graphically for fitting a suitable isotherm.

### Gasometric method

This technique gives accurate results compared to that of conventional weight loss method provided, the inhibitor does not react with hydrogen and the hydrogen penetration into the metal is small compared to the total volume of hydrogen gas. An improved design of the gasometric method as described elsewhere [16]. The specimen was suspended from the hook of the glass stopper and was introduced into the cell containing 100 mL of the experimental solution. The temperature was maintained constant throughout these experiments at 30°C and at constant atmospheric pressure. Volume measurements were made for a period of two hours in all the cases. From the volume of hydrogen gas liberated, the inhibition efficiency was calculated using the formula,

$$\text{Inhibition efficiency (\%)} = \frac{V_0 - V_1}{V_0} \times 100$$

Where, $V_0$ is the volume of hydrogen evolved in the absence of inhibitor and $V_I$ is the volume of hydrogen evolved in the presence of inhibitor.

**Potentiodynamic polarization studies**

Potentiodynamic polarization measurements were carried out using EG &G PAR potentiostat / galvanostat (Model - 173) analyzer a universal programmer in a conventional three – electrode glass cell. A platinum foil of surface area 2cm$^2$ was used as the auxiliary electrode and a saturated calomel electrode as the reference electrode. Both anodic and cathodic polarization curves were recorded in the absence and presence of an optimum concentration of the AML extract from a cathodic potential of –1900 mV to an anodic potential of – 1300 mV (vs SCE) at a sweep rate of 1mV per second. From the polarization curves, Tafel slopes, corrosion potential and corrosion current were calculated. The inhibitor efficiency was calculated using the formula;

$$IE\ (\%) = \frac{I_{Corr} - I^*_{Corr}}{I_{Corr}} \times 100$$

Where, $I_{corr}$ and $I^*_{corr}$ are corrosion current in the absence and presence of AML extract.

**Electrochemical impendance studies**

The electrochemical AC-impendance measurements were performed using EG & G Electrochemical impedance analyzer (model – 6310) with M38 software as described earlier [17]. Experiments were carried out at the open circuit potential for the frequency range of 100kHz to 10mHz. A plot of Z' vs Z" were made. From the plots, the charge transfer resistance ($R_t$) were calculated and the double layer capacitance were then calculated using the equation [18].

$$C_{dl} = 1 / 2\pi f_{max} R_t$$

Where $R_t$ is charge transfer resistance and $C_{dl}$ is double layer capacitance. The experiments were carried out in the absence and presence of an optimum concentration of inhibitor. The percentage of inhibition efficiency was calculated using the equation;

$$IE\ (\%) = \frac{R^*_t - R_t}{R_t^*} \times 100$$

Where $R_t$* and $R_t$ are the charge transfer resistance in the presence and absence of AML extract.

## RESULTS AND DISCUSSION

**Weight loss method**

The weight loss method was done with concentrations of AML leaves extract ranging from 200 to 1000 ppm (Table 1). Rise in concentration of AML extract extract, on the corrosion rate of aluminium in 1N NaOH solution was decreased and the inhibition efficiency increased from 70.5% to 87.6% upto 800ppm. Beyond this concentration, the corrosion inhibition efficiency was decreased from 87.6 to 84.7% . It indicates that 800ppm is the optimum concentration to get maximum corrosion protection for aluminium 1N NaOH using AML extract.

The effect of immersion time from 2 hours to 24 hours was also studied. The effect of immersion time on percentage inhibition efficiency of aluminium in 1N NaOH at 30°C in presence of an optimum concentration (800ppm) of AML extract is shown as Fig.1. It can be seen that the inhibition efficiency was found to decrease from 87.6% to 74.6%. Though 74.6% inhibition efficiency was obtained even at 24 hours of immersion time, the maximum inhibition efficiency was found at 2 hours. Hence, using weight loss method, it was found that AML extract acted as corrosion inhibitor at an optimum concentration of 800ppm for a period of 2 hours at 30°C.

**Table 1 Corrosion parameters obtained from weight loss measurements for aluminium in 1N NaOH containing different concentrations of AML extract**

| Conc. of AML extract (ppm) | Weigh Loss (gm) | Corrosion Rate (mmpy) | Inhibition Efficiency (%) | Surface Coverage (θ) |
|---|---|---|---|---|
| Blank | 0.3734 | 757.17 | ---- | ---- |
| 200 | 0.1100 | 223.05 | 70.5 | 0.71 |
| 400 | 0.0820 | 166.28 | 78.0 | 0.78 |
| 600 | 0.0620 | 125.72 | 83.4 | 0.66 |
| 800 | 0.0464 | 94.09 | 87.6 | 0.88 |
| 1000 | 0.0572 | 115.99 | 84.7 | 0.85 |

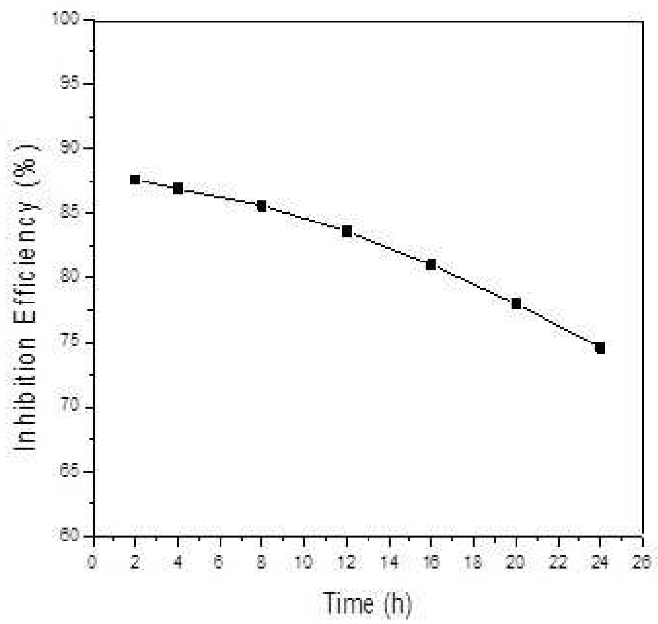


**Fig. 1 Effect of immersion time on percentage inhibition efficiency of aluminium in 1N NaOH at 30°C in presence of an optimum concentration (800ppm) of AML extract**

**Table 2 Inhibition efficiency obtained from gasometric measurements for aluminium in 1N NaOH containing various concentrations of AML extract at 30˚C**

| Conc. of AML Extract (% in v/v) | Volume of Hydrogen Gas evolved (mL) | *Inhibition Efficiency (%)* |
|---|---|---|
| Blank | 37.1 | -- |
| 200 | 11.0 | *70.3* |
| 400 | 8.0 | *78.4* |
| 600 | 6.1 | *83.6* |
| 800 | 4.6 | *87.6* |
| 1000 | 5.7 | *84.6* |

**Table 3 Potentiodynamic polarization parameters for aluminium in 1N NaOH in the absence and presence of an optimum concentration of AML extract**

| Conc. of *AML* Extract (ppm) | $E_{corr}$ (V vs SCE) | $I_{corr}$ ($mA/cm^2$) | Tafel Slope (mV/decade) | | Inhibition Efficiency (%) |
|---|---|---|---|---|---|
| | | | $b_a$ | $b_c$ | |
| Blank | -1.572 | 8.21 | 318 | 207 | __ |
| 800 | -1.571 | 1.00 | 302 | 200 | 87.8 |

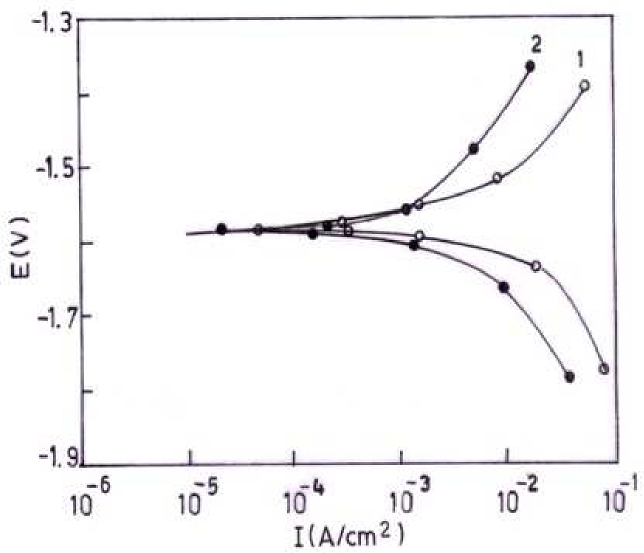


**Fig. 2 Potentiodynamic polarization curves for aluminium in 1N NaOH solution in the absence and presence of an optimum concentration of AML extract (1) Blank (2) 800ppm of AML extract**

**Gasometric method**

In the gasometric method, the increase in concentrations of AML extract from 200 to 1000 ppm,decreased the volume of hydrogen gas evolved from 11.0 to 4.6mL and hence the inhibition efficiency increased from 70.3 to 87.6% . Beyond this concentration, the inhibition efficiency decreased from 87.6 to 84.6% is shown in Table 2. Hence the optimum concentration of the extract was found to be 800ppm. It could be observed that AML extract has better ability to inhibit the corrosion of aluminium in alkaline solution.

**Potentiodynamic polarization method**

The potentiodynamic polarization parameters for aluminium in the absence and presence of an optimum concentration AML extract in 1N NaOH is given in Table 3 and its polarization curves are shown in Fig.2. It can be seen from the table that the corrosion potential was not shifted significantly in presence of the extract suggesting that AML extract control both anodic and cathodic reactions to inhibit the corrosion of aluminium by blocking active sites on the aluminium surface [19]. On the other hand, the corrosion current markedly decreased upon addition of the AML extract. With the addition of an optimum concentration of AML extract (800ppm), the maximum inhibition efficiency of 87.8 % was observed as in weight loss method. Hence, it is inferred that the inhibition action is of mixed type.

**AC impedance measurements**

The corrosion behaviour of aluminium in 1N NaOH in the absence and presence of AML extract was investigated by AC-impedance method to evaluate the charge transfer resistance ($R_t$) and double layer capacitance ($C_{dl}$). Through these parameters the inhibition efficiency was calculated. Fig.3 shows the impedance diagrams for aluminium in 1N NaOH in the absence and presence of an optimum concentration of AML extract and their corresponding impedance parameters are given in Table 4. It can be seen from the Nyquist plots that the curves are almost semicircular appearance followed by an inductive loop at the low frequency region . The semi circular nature of the Nyquist plot is due to the charger – transfer process, mainly control the corrosion of aluminium in 1N NaOH solution. Deviations of perfect circular shape are often referred to the frequency dispersion of interfacial impedance. This anomalous phenomenon may be attributed due to the inhomogeneity of the electrode surface arising from surface roughness or interfacial phenomena. The low frequency inductive loop is due to the growth and dissolution of the surface film [20]. In presence of AML extract, the value of $R_t$ increased and the value of $C_{dl}$ decreased. The decrease in $C_{dl}$ showed that the adsorption of the inhibitor took place on the aluminium surface in alkaline solution. The increase in the value of $R_t$ led to increase in the inhibition efficiency. The maximum $R_t$ value of 22.2 Ω $cm^2$ and the minimum $C_{dl}$ value of 17.32 µF/$cm^2$ was obtained at an optimum concentration of 800ppm of the AML extract, which gave the maximum inhibition efficiency of 88.5%. This result has good agreement with the results obtained from non-electrochemical methods.

**Table 4 Impedance parameters for the corrosion of aluminium in 1N NaOH in the absence and presence of an optimum concentration of AML extract at $30^0$C**

| Conc. of AML Extract (ppm) | $R_t$ (Ω $cm^2$) | $C_{dl}$ (µF/$cm^2$) | Inhibition Efficiency (%) |
|---|---|---|---|
| *Blank* | 2.56 | 82.12 | __ |
| 800 | 22.2 | 17.32 | 88.5 |

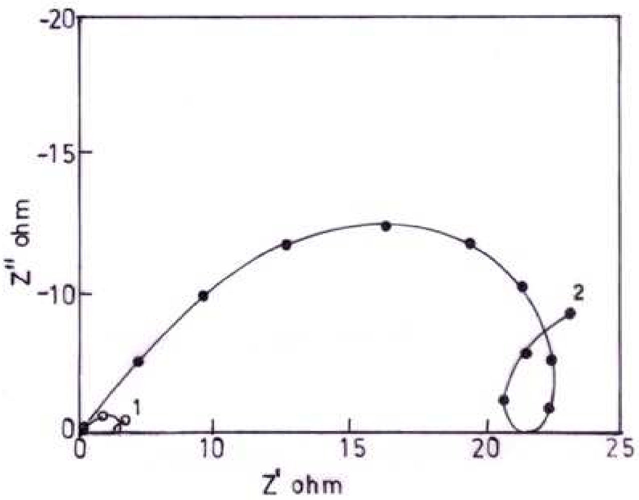


**Fig. 3 Impedance diagrams for aluminium in 1N NaOH solution in the absence and presence of an optimum concentration of AML extract (1) Blank (2) 800ppm of AML extract**

**Effect of Temperature**

The effect of temperature in the range of 30°C to 70°C on the corrosion behaviour of aluminium in 1N NaOH solution in the absence and presence of an optimum concentration of AML extract was studied using weight loss method as shown in Fig. 4 and its results are summarized in Table 5. It can be seen from the table that the increase in corrosion rate was more pronounced with the rise in temperature for the uninhibited alkaline solution than the inhibited solution suggesting that the extract adsorbed on the aluminium surface at all temperatures studied [21]. As temperature increased from 30°C to 70°C, the inhibition efficiency was found to slightly decrease from 87.6% to 82.0%. This shows that the adsorption of the extract on the aluminium may be due to physical adsorption.

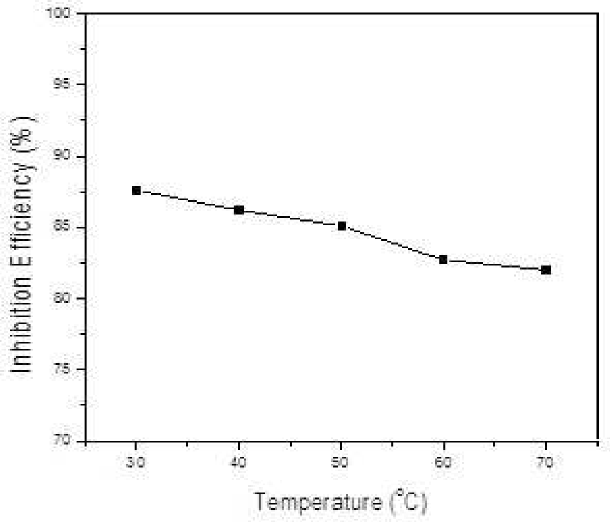


**Fig.4 Effect of temperature on the corrosion inhibition efficiency of aluminium in 1N NaOH in presence of an optimum concentration (800ppm) of AML extract**

**Table 5 Corrosion of aluminium in 1N NaOH solution in the absence and presence of an optimum concentration of AML extract at different temperatures obtained by weight loss method**

| System | Temperature (°C) | Corrosion Rate (mmpy) | Inhibition Efficiency (%) |
|---|---|---|---|
| Blank | 30 | 757.17 | --- |
| | 40 | 964.02 | --- |
| | 50 | 1172.42 | --- |
| | 60 | 1404.66 | --- |
| | 70 | 1697.76 | --- |
| 800 ppm of AML extract | 30 | 94.09 | 87.6 |
| | 40 | 133.03 | 86.2 |
| | 50 | 174.70 | 85.1 |
| | 60 | 243.00 | 82.7 |
| | 70 | 305.60 | 82.0 |

**Mechanism of corrosion inhibition**

The Arrehenius plot for aluminium immersed in 1N NaOH solution in the absence and presence of an optimum concentration (800ppm) of AML extract as shown in Fig. 5. It showing a straight line according to the Arrehenius equation and revealing the effect of temperature. The calculated values of activation energy (Ea), enthalpy of adsorption (ΔH), free energy of adsorption (ΔG°) and entropy of adsorption (ΔS**)** are shown in Table 6. The activation energy $E_a$ was found to be 17.42 KJ $mol^{-1}$ for blank and increased to 25.38 KJ $mol^{-1}$ in presence of AML extract in 1N NaOH suggesting that the adsorbed organic matter creates a physical barrier to charge and mass transfers, leading to reduction in corrosion rate. The higher value of $E_a$ in presence of the AML extract compared to that in the absence of the extract is due to physical adsorption.

**Table 6 Calculated values of activation energy (Ea), enthalpy of adsorption (ΔH), free energy of adsorption (ΔG°) and entropy of adsorption (ΔS) in the absence and presence of the optimum concentration of AML extract**

| System | Temp. (K) | Ea (KJ $mol^{-1}$) | ΔG° (KJ$mol^{-1}$) | ΔH (KJ$mol^{-1}$) | ΔS (KJ$mol^{-1}$) |
|---|---|---|---|---|---|
| Blank | 303 | 17.42 | --- | 14.90 | --- |
| | 313 | | --- | 14.81 | --- |
| | 323 | | --- | 14.73 | --- |
| | 333 | | --- | 14.65 | --- |
| | 343 | | --- | 14.56 | --- |
| 800 ppm of AML extract | 303 | 25.38 | -15.61 | 22.86 | 0.127 |
| | 313 | | -15.80 | 22.77 | 0.123 |
| | 323 | | -16.04 | 22.69 | 0.120 |
| | 333 | | -16.07 | 22.61 | 0.116 |
| | 343 | | -16.41 | 22.52 | 0.114 |

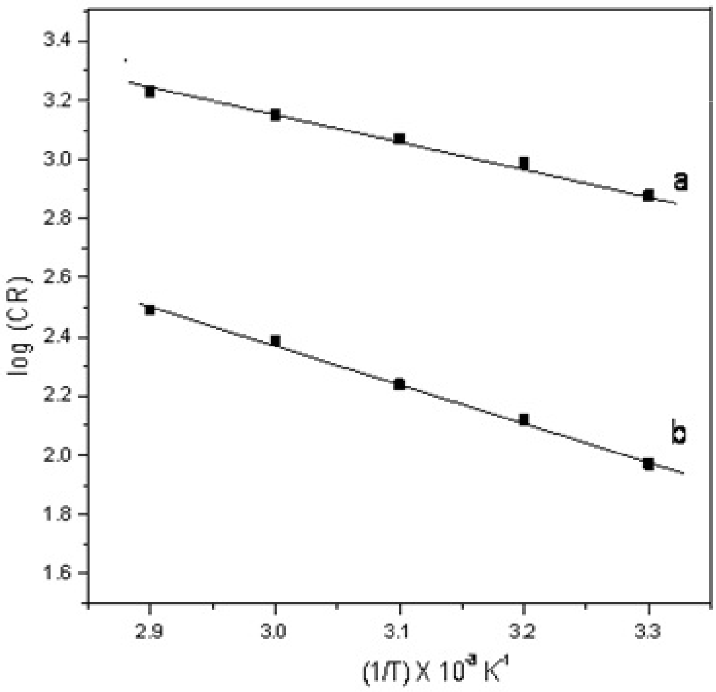


**Fig. 5 Arrhenius plots for aluminium immersed in 1N NaOH solution in the absence and presence of an optimum concentration ( 800ppm) of AML extract**

The negative sign of free energy of adsorption indicates that the adsorption of AML extract on aluminium surface is a spontaneous process [22]. In this study, the ΔG° values are in the range of -15.61 KJ $mol^{-1}$ to -16.41 KJ $mol^{-1}$. As the values of free energy of adsorption are less than -20 KJ $mol^{-1}$, where the mode of inhibition is due to physisorption. The positive values of enthalpy of adsorption (ΔH) shows that the reaction was endothermic and the adsorption of the inhibitor on the aluminium surface took place easily. Entropy of adsorption (ΔS) remains a positive value and this reflects the formation of an ordered stable layer of the inhibitor molecule on the aluminium. Moreover, the positive values of entropy indicates that the reaction was spontaneous and feasible.

The corrosion of aluminium in NaOH solution is a heterogonous one, composed of anodic and cathodic reactions. Based on this, the kinetic analyses of the data studied for a period of 2 hours immersion time were considered. The major constituent present in the leaves of aegle marmelos extract contains the neutral alkaloid Aegelin [23,24]. Inspection of the chemical structure of wedelactone and ecliptine revealed that the compound adsorb on the aluminium surface via the lone pair of electrons present on their oxygen atoms. The adsorption of such compounds on the metal surface made a barrier for charge and mass transfers leading to a decrease in the interaction of aluminium with the corrosive environment. As a result, the corrosion rate of aluminium decreased. The formation of film layer, prevented the corrosion of aluminium. The use of adsorption isotherm provides useful insight onto the corrosion inhibition mechanism. The adsorption of different concentrations of AML extract on the surface of aluminium in 1N NaOH solution followed Langmuir adsorption isotherm (Fig. 6)

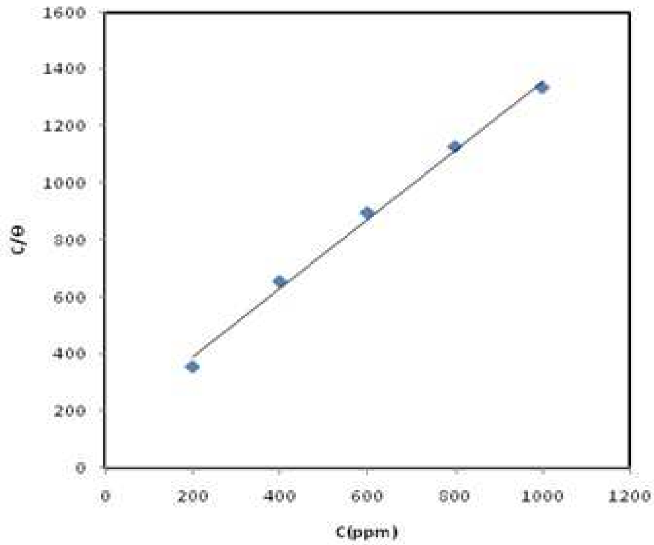


**Fig. 6 Langmuir adsorption isotherm plot for the adsorption of various concentrations of AML extract on the surface of aluminium in 1N NaOH solution**

**Surface Analysis**

Surface examination of the aluminium specimens was made using JEOL-Scanning electron microscope (SEM) with the magnification of 1000X is shown in Fig.7 (a & b). The SEM studies showed that the inhibited aluminium surface was found smother than the inhibited surface due to the formation of protective film on the inhibited aluminium surface.

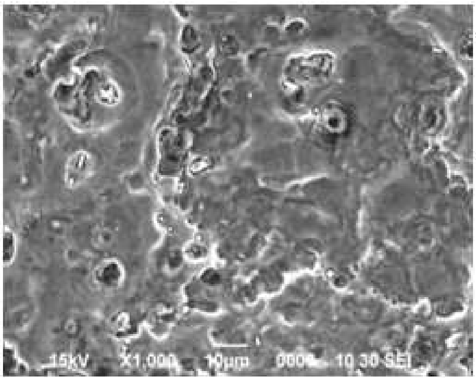

**Fig.7 (a) SEM Photograph of aluminium immersed in 1N NaOH solution (blank)**

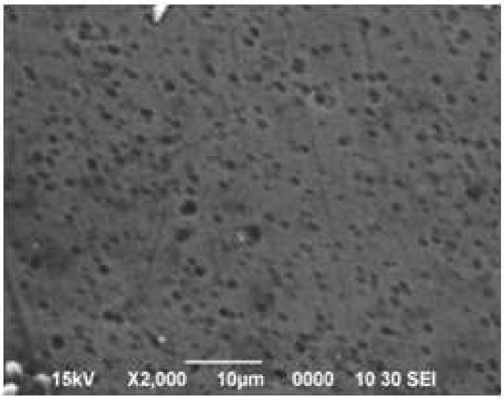


**Fig. 7 (b) SEM Photograph of aluminium immersed in 1N NaOH solution containing an optimum concentration (800ppm) of AML extract**

## CONCLUSION

The following conclusions are drawn from the above studies:

- The aegle marmelos leaves (AML) extract perform well in 1N NaOH solution and inhibit the corrosion of aluminium at an optimum concentration of 800ppm.
- The AML extract control both anodic and cathodic reactions by blocking the active sites of aluminium surface and thus the inhibitor of mixed type.
- The AML extract inhibit the corrosion of aluminium in 1N NaOH solution by strong adsorption of its chemical constituents on the aluminium surface obeys Langmuir adsorption isotherm.
- The SEM studies showed that the inhibited aluminium surface was found smother than the inhibited surface due to the formation of protective film on the inhibited aluminium surface.

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