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Green Approach To Corrosion Inhibition Of Aluminium By *Senna Auriculata* Leaves Extract In 1 N NaOH Solution

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ABSTRACT

Corrosion inhibition of aluminium in 1N NaOH by leaves extract of *Senna auriculata* (SL) was studied using chemical and electrochemical techniques at 30-60°C. It was found that the leaves of *Senna auriculata* inhibited the base induced corrosion of aluminium.The inhibition efficiency increasing with increase in concentration of SL extract and reached the maximum of 76.2% that decreases with rise in temperature. The adsorption of SL on metal surface obeys Langmuir adsorption isotherm. Polarization measurements showed that SL extract acted as mixed type inhibitor. The surface morphology of aluminium in the absence and presence of SL extract in 1N NaOH solution, was studied using scanning electron microscopy. Results obtained from chemical and electrochemical techniques were in good agreement. Thermodynamic parameters such as activation energy and free energy of adsorption was also calculated to explain the inhibition mechanism.

Keywords: *Aluminium corrosion, Corrosion inhibitors, Senna auriculata leaves extract, Langmuir adsorption isotherm, Mixed type inhibitor.*

INTRODUCTION

Corrosion involves the movement of metal ions into the solution at active areas (anode), passing of electrons from the metal to acceptors at less active areas (cathode), an ionic current in the solution and an electric current in the metal. The cathodic process requires the presence of an electron acceptor such as oxygen or oxidizing agents or hydrogen ions (1-5). Aluminium and its alloys are extremely used in numerous industrial and engineering applications. The corrosion resistance is due to the initial formation of a compact and adherent passive oxide film on the exposed surfaces. However, in the presence of insidious ions such as chloride ions, the protective oxide film can be locally destroyed, initiating metal dissolution. Again, the oxide film is amphoteric and hence dissolves readily in acidic solutions (6).The corrosion behavior of pure aluminium and its alloys in alkaline solution have been extensively studied in the development of aluminium anodes for aluminium/air batteries [7]. Aluminium corrosion within the battery cause many problem because in most aqueous solutions, the anodic overvoltage for the dissolution of aluminium is very high[8]. 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.

The use of chemical inhibitors has been limited because of the environmental regulations, and hence plant extracts have become important because they are environmentally acceptable, readily available and renewable source for a wide range of needed inhibitors [9]. For instance, henna leaves [10] gossipium higgutum [11], gum arabic [12] phyllanthusamarus [13], ipomoea involucrate [14], hibiscus sabdoriffa [15] adathoda vasica [16] damisissa [17], brahmi [18], pipali leaves [19] etc., have been studied as effective corrosion inhibitors for aluminium in alkaline medium. *Senna auriculata* commonly known as tanner's cassia, also known as "avaram" in Tamil language is a shrub belongs to the Caesalpiniaceae family. It occurs in the dry regions of India and Sri Lanka. The shrub is specially famous for its attractive yellow flowers which are used in the treatment of skin disorders and body odour.It is widely used in traditional medicine for rheumatism, conjunctivitis and diabetes. It has many medicinal properties.It was also observed that flower and leaf extract of *Senna auriculata* shown to have antipyretic activity. Owing to its reputed medicinal value, the plant is now widely used in India and Sri Lanka as part of the Ayurvedic system of medicine [20].

This paper reports the influence of *Senna auriculata* (SL) extract on the corrosion of aluminium in 1N alkaline solution by weight loss, gasometric, electrochemical 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 of film on the surface of aluminium.

EXPERIMENTAL

Material preparation

Aluminium strips of 4.5cm × 2cm × 0.2cm containing > 99.9 % purity was used for weight loss and gasometric 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 $0.5 cm^2$ was used for electrochemical polarization studies and AC impedance measurements. Analar grade NaOH and double distilled water were used to prepare the solutions.

Preparation of leaves of *Senna auriculata* leaves (*SL*) extract

The *Senna auriculata* leaves (Aavaram) were collected and cut into small pieces. They were dried in an air oven at 80°C for 2 hrs. The dried leaves were ground well into powder. From this, 10g of the sample was refluxed in 100mL of 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 dried compound [21]. The *SL* extract concentrations of 300 to 900 ppm were prepared using 1N NaOH solution.

Weight loss studies

Weight loss measurements were carried out as described elsewhere [22]. Aluminium specimens were immersed in 100ml of inhibited and uninhibited solutions for 2 hours at 30°C. The corrosion rate (mmpy) and the inhibition efficiency were calculated using the described elsewhere. Weight loss measurements were also performed at various immersion time from 2 hours to 10 hours for the best concentration of the *SL* 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 [22];

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

Where, WB is the weight loss in the absence of the SL extract, WI is the weight loss in the presence of the SL 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 [23]. 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;

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

Where, V_0 is the volume of hydrogen evolved in the absence of SL extract and V_1 is the volume of hydrogen evolved in the presence of SL extract.

Electrochemical polarization studies

Electrochemical polarization measurements were carried out using Electrochemical analyzer (BioLogic, VSP, France) 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 various concentrations of the *SL* 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 [24];

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

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

Electrochemical impedance studies

The electrochemical AC-impedance measurements were performed using Electro-chemical analyzer (BioLogic, VSP, France) as described earlier. Experiments were carried out at the open circuit potential for the frequency range of 100kHz to 1mHz.A plot of Z' vs Z" were made. From the plots, the charge transfer resistance (R_t) were calculated and the double layer capacitance (C_{dl}) were then calculated using the following equation [25];

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

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 various concentrations of *SL* inhibitor. The percentage of inhibition efficiency was calculated using the equation [26];

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

Where, R_t* and R_t are the charge transfer resistance in the presence and absence of SL extract.

Surface examination studies

The aluminium specimens were immersed in 1N NaOH in the absence and presence of the best concentration of *SL* extract for 2 hours at 30°C. After 2 hours, the specimens were taken out, dried and kept in desiccators. The protective film formed on the surface of aluminium was confirmed by SEM studies with the magnification of 1000x.

RESULTS AND DISCUSSION

Weight loss method

The weight loss method was done with concentrations of *SL* extract ranging from 300 to 900ppm (Table 1). Rise in concentration of *SL* extract decreased the corrosion rate of aluminium in 1N NaOH solution and increased the inhibition efficiency, increased from 53.8 % to 76.2 % upto 900ppm.It indicates that 900ppm is the best concentration to get maximum corrosion protection for aluminium in 1N NaOH using *SL* extract. This trend may result from the fact that adsorption is enhanced with increase in concentration of *SL* extract. As a result, more inhibitor molecules are adsorbed on the metal surface reduces the surface area available for the attack of the aggressive OH^- ions from the alkaline solution for corrosion. The effect of inhibition efficiency with various concentrations of SL extract on aluminium in 1N NaOH is shown in Fig.1.

The effect of immersion time from 2 to 10 hours was also studied. The inhibition efficiency was found to decrease from 76.2 % to 72.0 %.The slight decrease in inhibition efficiency at longer immersion time is due to an increase in cathodic or hydrogen evolution kinetics or decreasing strength of adsorption (shifting adsorption-desorption equilibrium toward desorption). The effect of immersion time on percentage inhibition efficiency of aluminium in 1N NaOH at 30^0C in presence of the best concentration (900 ppm) of *SL* extract is given in Table-2 (Fig.2). Though 72.0 % inhibition efficiency was obtained even at 10 hours of immersion time, the maximum inhibition efficiency was found at 2 hours. Hence, using weight loss method, it was found that *SL* extract acted as corrosion inhibitor for aluminium in 1N NaOH medium at the best concentration of 900 ppm for a period of 2 hours at 30^0C.

Table 1 Corrosion parameters obtained from weight loss measurements for aluminium in 1N NaOH solution containing various concentrations of SL extract

Conc. of SL Extract (ppm)	Weight Loss (gm)	Corrosion Rate (mmpy)	Inhibition Efficiency (%)	Surface Coverage (θ)
Blank	0.2424	436.92	-	-
300	0.1120	201.88	53.8	0.538
500	0.0844	152.13	65.2	0.652
700	0.0664	119.68	72.6	0.726
900	0.0576	103.82	76.2	0.762

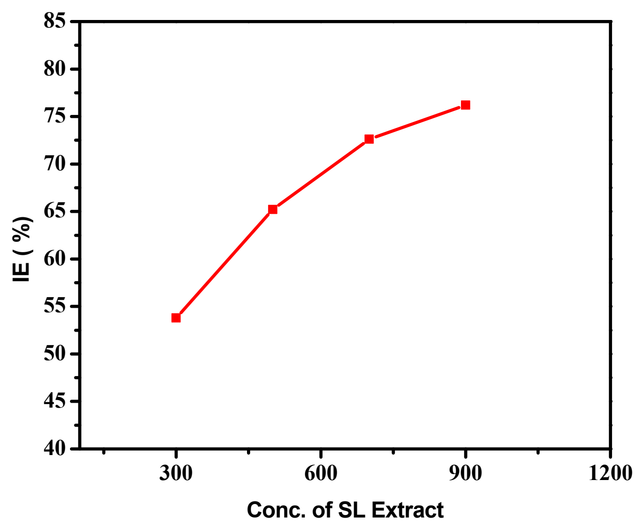

Fig.1 Inhibition efficiency with various concentrations (ppm) of SL extract on aluminium in 1N NaOH at 30°C

Table 2

Effect of immersion time on percentage inhibition efficiency of aluminiumin 1N NaOH at 30°C in presence of the best concentration (900ppm) of SL extract

System	Weight Loss (gm)					IE (%)				
	Time (hrs)					Time (hrs)				
	2	4	6	8	10	2	4	6	8	10
Blank	0.2424	0.3482	0.5482	0.7262	0.8560	-	-	-	-	-
900 ppm of SL extract	0.0577	0.0850	0.1403	0.1946	0.2397	76.2	75.6	74.4	73.2	72.0

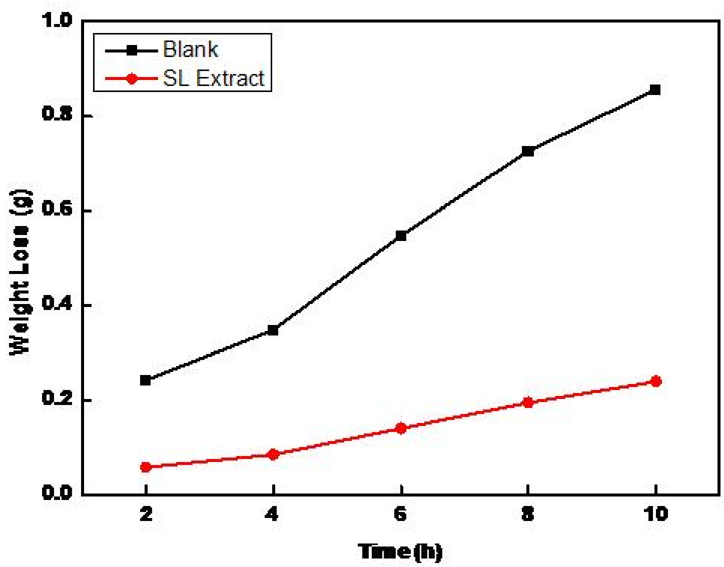

Fig.2 Effect of immersion time on percentage inhibition efficiency of aluminium in 1N NaOH at 30°C in presence of the best concentration (900ppm) of SL extract

Gasometric method

In the gasometric method, the increase in concentrations of *SL* extract from 300 to 900ppm decreased the volume of hydrogen gas evolved from 26.4 to 6.3 mL and hence the inhibition efficiency increased from 52.6 to 76.0 % is shown in Table 3. Hence the best concentration of the *SL* extract was found to be 900ppm. It could be observed that *SL* extract has better ability to inhibit the corrosion of aluminium in alkaline solution.

Table 3 Inhibition efficiency obtained from gasometric measurements for aluminium in 1N NaOH containing various concentrations of *SL* extract at 30˚C

Conc. of SL Extract (ppm)	Volume of Hydrogen Gas evolved (mL)	Inhibition Efficiency (%)
Blank	26.4	-
300	12.5	52.6
500	9.3	64.8
700	7.3	72.2
900	6.3	76.0

Electrochemical polarization method

The electrochemical polarization parameters for aluminium in the absence and presence of various concentrations *SL* extract in 1N NaOH is given in Table 4 and their polarization curves are shown in Fig.3. It can be seen from the table that the corrosion potential was not shifted significantly in presence of the extract suggesting that *SL* extract control both anodic and cathodic reactions to inhibit the corrosion of aluminium by blocking active sites on the aluminium surface [27]. On the other hand, the corrosion current density was markedly decreased upon the addition of the *SL* extract in 1N NaOH. With the addition of the best concentration of *SL* extract (900ppm), the maximum inhibition efficiency of 75.0 % was observed as in weight loss method. Hence, it is inferred that the inhibition action is of mixed type.

Table 4 Electrochemical polarization parameters for aluminium in 1N NaOH solution in the absence and presence of various concentrations of SL extract

Conc. of SL extract (ppm)	E_{corr} (V vs SCE)	I_{corr} (mA/cm^2)	Tafel Slope (mV/decade)		Inhibition Efficiency (%)
			b_a	b_c	
Blank	-1.584	4.46	182	318	---
300	-1.582	2.131	174	296	52.2
500	-1.580	1.606	168	286	64.0
700	-1.570	1.302	172	296	70.8
900	-1.560	1.115	170	292	75.0

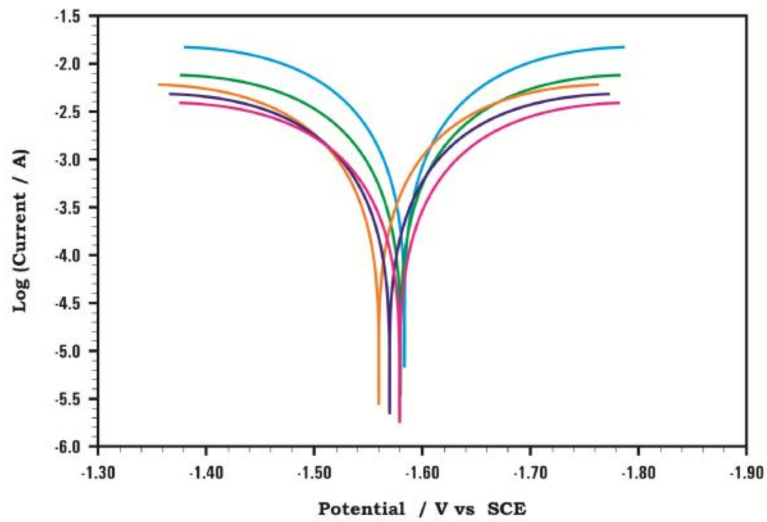

Fig. 3 Electrochemical polarization curves for aluminium in 1N NaOH solution in the absence and presence of various concentrations of SL extract

AC- impedance measurements

Fig.4 shows the impedance diagrams for aluminium in 1N NaOH in the absence and presence of various concentrations of *SL* extract and their corresponding impedance parameters are given in Table 5. It can be seen from the figure that the obtained Nyquist plots are almost semicircular in nature followed by an inductive loop at the low frequency region. The semicircular nature of the Nyquist plot is due to the charge - transfer process, mainly controls the corrosion of aluminium 1N NaOH solution. Deviations of perfect circular shape are often referred to the frequency dispersion of interfacial impedance. This anomalous phenomenon may be attributed 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 [8]. In fact, the presence of SL extract enhanced the value of R_t in alkaline solution and the values of double layer capacitance are brought down to the maximum extent in presence of inhibitor. 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 with the inhibitor led to increase in the inhibition efficiency. The maximum R_t value of 18.14 Ω cm^2 and minimum C_{dl} value of 4.18 μF/ cm^2 was obtained at the best concentration (900 ppm) of the extract gave a maximum inhibition efficiency of 75.1%. This result has good agreement with the results obtained from non-electrochemical methods (weight loss method and gasometric method) and electrochemical polarization method.

Table 5 Impedance parameters for the corrosion of aluminium in 1N NaOH in the absence and presence of various concentrations of *SL* extract at 30^{0}C

Conc. of SL Extract (ppm)	R_t (Ω cm^2)	C_{dl} (μF/cm^2)	Inhibition Efficiency (%)
Blank	4.52	108.36	---
300	9.54	23.64	52.6
500	12.68	18.40	64.3
700	16.00	8.36	71.7
900	18.14	4.18	75.1

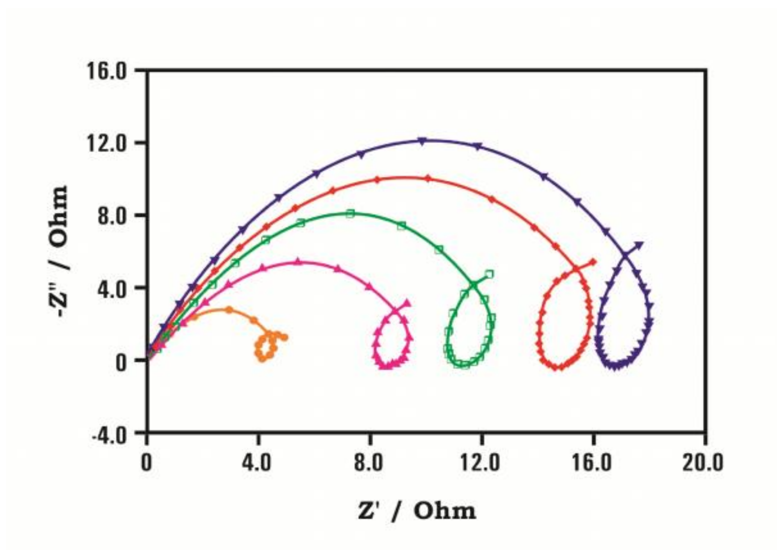

Fig. 4 Impedance diagrams for aluminium in 1N NaOH solution in the absence and presence of various concentrations of *SL* extract

Effect of Temperatures

The effect of temperature in the range of 30°C to 60°C on the corrosion behaviour of aluminium in 1N NaOH solution in the absence and presence of the best concentration of *SL* extract was studied using weight loss method as shown in Fig.5 and its results are summarized in Table 6.

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. As the temperature increased from 30°C to 60°C, the inhibition efficiency was found to slightly decrease from 76.2 % to 69.6 %. This shows that the adsorption of the extract on the aluminium may be due to physical adsorption.

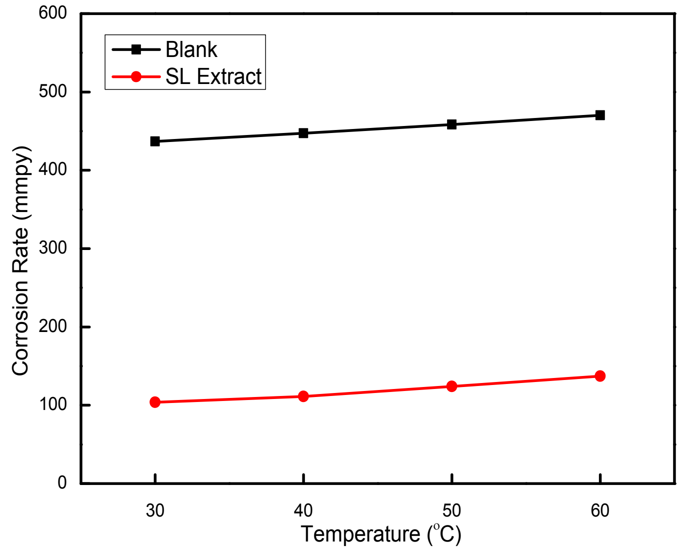

Fig.5 Effect of temperature on the corrosion inhibition efficiency of aluminium in 1N NaOH in presence of the best concentration (900ppm) of *SL* extract

Table 6 Corrosion of aluminium in the absence and presence of the best concentration of SL extract (900 ppm) in 1N NaOH at different temperatures obtained by weight loss method

System	Temperature (°C)	Corrosion Rate (mmpy)	Inhibition Efficiency (%)
Blank	30	436.92	---
	40	447.46	---
	50	458.51	---
	60	470.14	---
900 ppm of SL extract	30	103.82	76.2
	40	111.28	74.8
	50	123.94	72.3
	60	137.18	69.6

Mechanism of corrosion inhibition

The Arrhenius plot for aluminium immersed in 1N NaOH solution in the absence and presence of the best concentration (900 ppm) of *SL* extract as shown in Fig. 6. It showing a straight line according to the Arrehenius equation and revealing the effect of temperature. The calculated values of activation energy (E_a) and free energy of adsorption ($\Delta G°$) are shown in Table 7. The activation energy E_a was found to be 1.87 kJ mol^{-1} for blank and increased to 5.47 kJ mol^{-1} in presence of *SL* 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 *SL* extract compared to that in the absence of the extract is due to physical adsorption [17, 28].

Table 7 Calculated values of activation energy (E_a) and free energy of adsorption (ΔG^o) in the absence and presence of the best concentration (900 ppm) of *SL* extract

System	E_a (kJ/mol)			ΔG^o (kJ/mol)			
	Temperature (ºC)			Temperature (ºC)			
	30-40	40-50	50-60	30	40	50	60
Blank	1.87	2.05	2.23	-	-	-	-
900 ppm of SL extract	5.47	9.05	9.08	-13.31	-13.56	-13.64	-13.70

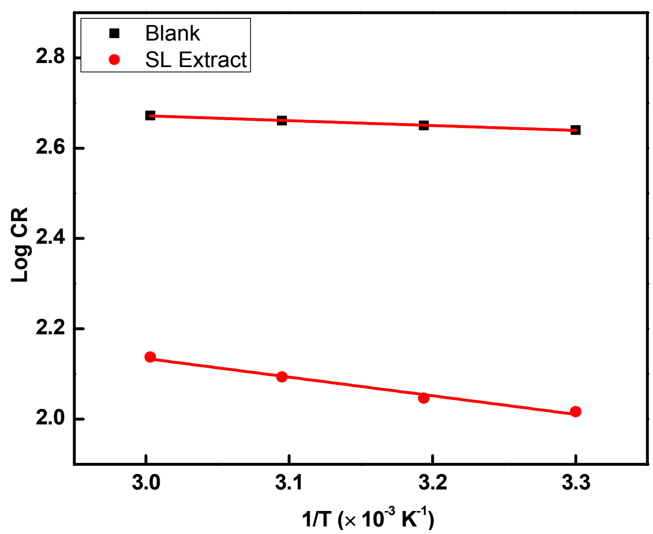

Fig. 6 Arrhenius plots for aluminium immersed in 1N NaOH solution in the absence and presence of the best concentration (900ppm) of *SL* extract

The negative sign of free energy of adsorption indicates that the adsorption of SL extract on aluminium surface is a spontaneous process [29]. It is well known that the value of ΔG° around -13.31 kJ mol^{-1} or lower are consistent with the electrostatic interaction between organic charged molecules and the charged metal (physisorption) and those around -40 kJ mol^{-1} or higher involved charge sharing or transfer from the organic molecules to the metal surface to form a co-ordinate type of bond (chemisorption) [30]. In this case, the ΔG° values were in the range -13.31 to -13.70 kJ mol^{-1} and hence the adsorption could be physisorption.

From the literature survey, it was found that luteolin, quercetin and kaempferol are the principal alkaloids present in the extract of SL [31]. The inhibition of SL extract may be due to the presence of the major alkaloids luteolin, quercetin and kaempferol which contain hydroxyl groups. The structures of luteolin, quercetin and kaempferol are shown in Fig.7. The inhibition mechanism involved in this is due to the adsorption of inhibitor SL extract, on the surface of the aluminium and thus retarding the corrosion.

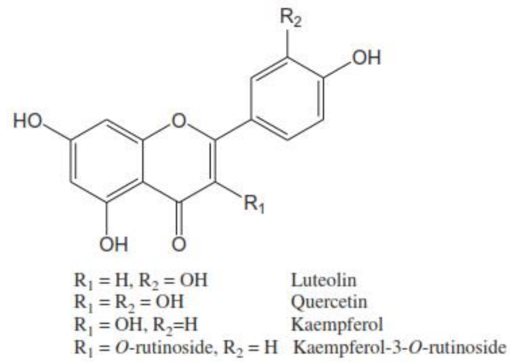

Fig.7 Structures of Luteolin , Quercetin and Kaempferol

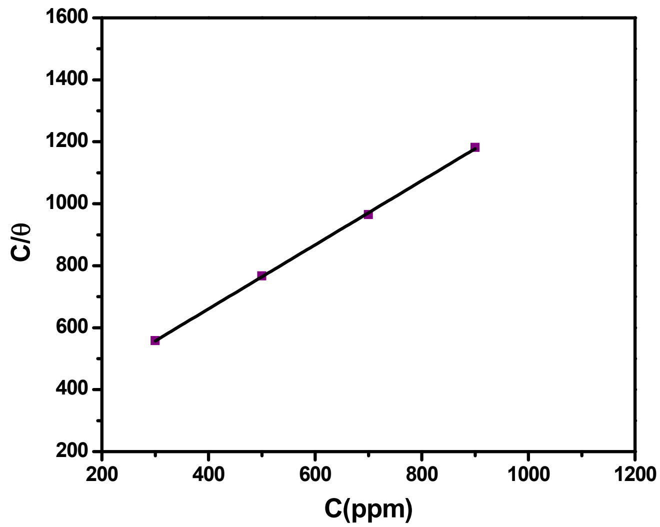

Fig.8 Langmuir adsorption isotherm plot for the adsorption of various concentrations of SL extract on the surface of aluminium in 1N NaOH solution

The adsorption of these compounds on the aluminum surface made a barrier for mass and charge transfers. This situation led to the protection of aluminium surface from the action of aggressive ions of the alkaline solution. The degree of protection increased with surface coverage by the adsorbed molecules. As the extract concentration increased, the number of adsorbed molecules on the surface increased. Surface coverage (θ) which was estimated from the inhibition efficiency values using weight loss method could be used to represent the fraction of the surface occupied by the adsorbed molecules. The values of surface coverage (θ) for various concentrations of SL extract are given in Table 1. The use of adsorption isotherm provides useful insight into the corrosion inhibition mechanism. A plot of C/θ versus C gave a straight line with unit slope suggests that the adsorption of various concentrations of SL extract on the surface of aluminium in 1N NaOH solution follows Langmuir adsorption isotherm (Fig. 8).

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Surface Analysis

Surface examination of the aluminium specimens was made using JEOL-scanning electron microscope (SEM) with the magnification of 1000x. The aluminium specimens after immersion in 1N NaOH solution for 2 hours at 30°C in the absence and presence of the best concentration of the SL extract were taken out, dried and kept in a dessicator. Their surface was examined by SEM studies are shown as Fig.9 (a & b). In the absence of SL extract, the corroded aluminium surface with etched grain boundaries are clearly seen. In the presence of SL extract, almost 76.2 % of protected aluminium surface are seen.

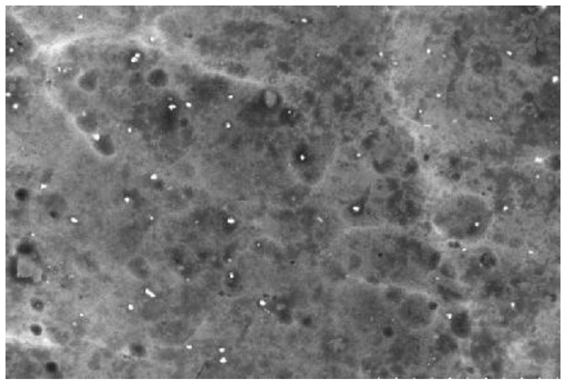

Fig.9 (a) SEM Photograph of aluminium immersed in 1N NaOH solution (blank)

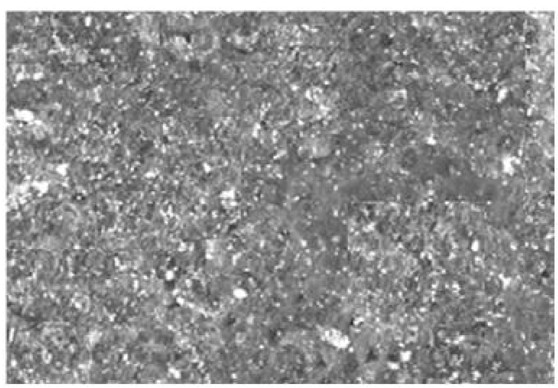

Fig.9 (b) SEM Photograph of aluminium immersed in 1N NaOH solution containing the best concentration (900ppm) of SL extract

CONCLUSIONS

Based on the results of investigation, the following conclusions are drawn:

- ✓ The Senna auriculata leaves (SL) extract is a good eco-friendly green inhibitor for the corrosion of aluminium in 1N NaOH solution.
- ✓ Inhibition efficiency of SL extract increases with increase in the concentration of SL extract and inhibit the corrosion of aluminium at the best concentration of 900ppm.
- ✓ Inhibition efficiency of SL extract decreases with increase in temperature.
- ✓ Electrochemical polarization curves proved that SL extract act as mixed type inhibitor by inhibiting both anodic and cathodic reactions to some extent.
- ✓ The adsorption of SL extract on the surface of aluminium follows Langmuir adsorption isotherm.The negative values of free energy of adsorption (ΔG°) indicate that the adsorption process is spontaneous and physically adsorbed on the aluminium surface.
- ✓ Surface photographs showed a good surface coverage on aluminum surface after being treated with SL extract.

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