

Studies on acid corrosion of aluminium by a naturally occurring polymer (Xanthan gum)

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Abstract - The corrosion inhibition of aluminium in hydrochloric acid solution by xanthan gum has been studied using weight loss measurements technique. Results show that xanthan gum inhibited aluminium dissolution in 0.5 M HCl solution and the inhibition was found to increase with increasing concentration of the inhibitor. Also, inhibition efficiency increased with rise in temperature. The activation energy, E_a as well as other thermodynamic parameters (Q_{ads} , ΔG_{ads} , ΔH° and ΔS°) for the inhibition process was calculated and results obtained corroborated the proposed inhibition mechanism. The corrosion inhibition of aluminium in 0.5 M HCl solution by xanthan gum has been suggested to occur by adsorption at the metal/solution interface following Temkin adsorption isotherm. Polarization measurements indicate that xanthan gum is a mixed type inhibitor with pronounced cathodic inhibitive effect.

Index Terms - Acid inhibition, Adsorption, Aluminium, Polymer, Temkin isotherm, Weight loss, Xanthan gum.

1 INTRODUCTION

The study of corrosion of metals and their inhibition has been a subject of immense technological importance, due to the extensive industrial applications of metals. In particular, aluminium and its alloys represent an important category of industrial metals due to their specific strength, thermal and electrical conductivities, heat and light reflectivity [1], excellent formability and their passive behaviour [2].

However, during service, aluminium-based components come in contact with some aggressive solutions such as hydrochloric acid, where they are used for acid pickling of aluminium, cleaning of oil refinery equipment and for chemical and electrochemical etching [3]. These activities normally lead to substantial loss of metal to corrosion. Consequently, the integrity of the aluminium's protective layer is affected, which leads to breakdown of the surface film in such environment and exposes bare aluminium surface sites to the corrosive, thereby accelerating the corrosion reaction [2,4].

Over the years, there has been increasing efforts to ameliorate the dissolution of aluminium in acidic solutions and hence preserve the integrity of the metal in the aggressive solutions [5-7]. The most useful approach is to effectively isolate the metal surface from the corrosion agents. This could effectively be achieved by use of corrosion inhibitors [8-11]. A corrosion inhibitor is a chemical substance or combination of substances that when present in the aggressive environment, prevents, or reduces corrosion rate of a metal exposed to that environment, without a significant reaction with components of the environment [12,13]. The corrosion inhibitor can act in a number of ways; it may restrict the rate of the anodic process or the cathodic process by simply blocking active sites on the metal surface [14,15]. Alternatively, it may act by increasing the potential of the metal surface so that the metal enters the

passivation region where a natural oxide film forms. A further mode of action of some corrosion inhibitors is that the inhibiting compound contributes to the formation of thin layer at the surface which stifles the corrosion process [16].

Among the numerous corrosion inhibitors available, organic compounds containing polar functions such as nitrogen, sulphur and/or oxygen in a conjugated system have been reported to exhibit good inhibiting effects on metal corrosion *via* adsorption at the metal/aggressive solution interface [2, 3, 17-19]. The efficiency of inhibition depends on the mechanical, structural and chemical characteristics of the adsorbed layers formed under particular conditions [14, 20].

Some investigations have in recent time been made into the corrosion inhibiting effect of some polymers on aluminium dissolution, which have been found to generally exhibit good inhibition efficiencies [21-25]. This is because of the ease with which they form protective complexes with metal ions, through their functional groups. The formed complexes occupy a large surface area, thereby blanketing the surface and protecting the metal from corrosive agents [22, 26]. The inhibitive power of polymers is related structurally to the cyclic rings, heteroatoms (oxygen and nitrogen) that are the major active centres of adsorption [23, 25, 27].

As a contribution to the current exploitation of organic polymeric corrosion inhibitors, the present study investigates the inhibiting effect of xanthan gum (Fig. 1) on aluminium corrosion in hydrochloric acid solution using weight loss measurements technique. The effect of temperature on the inhibition efficiency has also been studied. Kinetic and activation as well as adsorption parameters that govern metal corrosion have been evaluated.

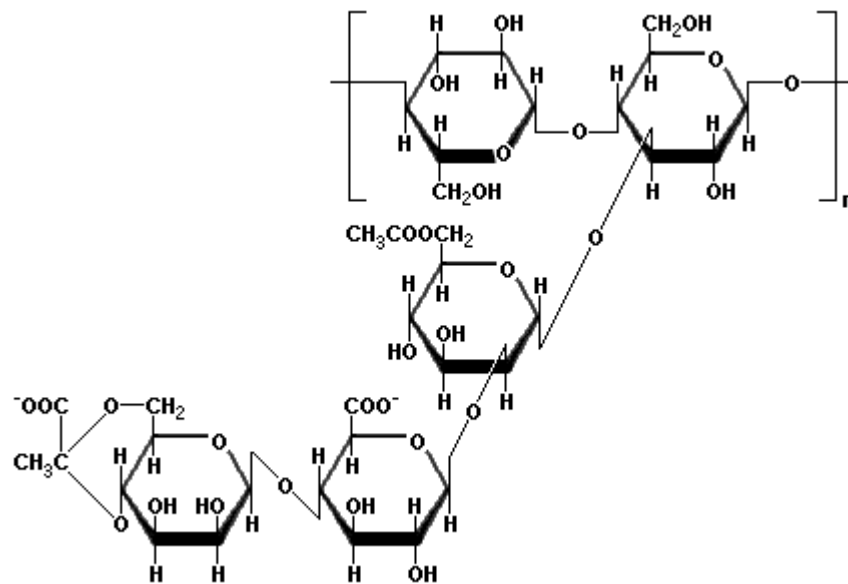


Fig. 1. Chemical structure of xanthan gum

2 EXPERIMENTAL TECHNIQUE

The weight loss measurements according to standard method reported by some authors [28-30] were carried out in a 250 ml capacity glass beaker. The blank acid test solution was prepared using serial dilution principle. The solutions containing xanthan gum additive were prepared in five different concentrations - 1.0, 2.0, 3.0, 4.0 and 5.0 g/l. The xanthan gum used was obtained from Sigma Aldrich chemical company. The hydrochloric acid was of BDH analytical reagent chemical.

The study was carried out first at ambient temperature, 28±1 °C and later in a thermostatic water bath equilibrated at 40, 50 and 60 °C. The aluminium samples used were press-cut mechanically into coupons of dimension (length = 4 cm, width = 3 cm and thickness = 0.045 cm) and used without further polishing but were degreased in acetone. The aluminium used has the following chemical composition (wt %): Si, 0.842; Fe, 0.898; Cu, 0.028; Mn, 0.081; Mg, 0.026; Zn, 0.010; Ti, 0.015; Cr, 0.006; Ni, 0.003; V, 0.009 and balance Al. The coupons were initially weighed and suspended in 200 ml of the test solution and kept under aerated condition and were monitored at 24-h interval progressively for 120-h (5 days). For the temperature study, the experiment was conducted for 3-hs. At the end of the tests, the coupons were taken out, washed with double distilled water, and then with acetone, dried and then reweighed using an analytical balance (precision = ±0.001 mg).

Three measurements were performed in each case and the mean value of the weight loss has been reported. The corrosion rate (C_r) of the aluminium in the test solutions were calculated from the expression

$$C_r = \left(\frac{87,600 \Delta W}{\rho A t} \right) \quad (1)$$

where ΔW is the weight loss (g), ρ the density of the aluminium (g/cm³), A the surface area and t the exposure time.

From the values of corrosion rate in the absence (C_r^{blk})

and presence of xanthan gum (C_r^{xg}), the inhibition efficiency ($IE\%$) over the exposure time period were calculated according to the following equation

$$IE\% = \left(1 - \frac{C_r^{xg}}{C_r^{blk}} \right) \times 100 \quad (2)$$

Electrochemical polarization test was carried out in a conventional three-electrode configuration with graphite rod as counter electrode and saturated calomel electrode (SCE) as the reference electrode. The working electrode (1cm² surface area) was first immersed in the test solution and after establishing a steady state OCP (open circuit potential), the electrochemical polarization measurements were carried out in a VERSASTAT3 computer controlled electrochemical workstation. The potentiodynamic polarization curves were obtained in the potential range -250 mV to +1600 mV with a sweep rate of 0.5 mV/s.

3 RESULTS AND DISCUSSION

3.1 Weight loss and corrosion rates

The free corrosion of aluminium in 0.5 M HCl, characterized by reduction in the thickness of the metal samples resulting from weight loss and corrosion rates in the absence and presence of additive was investigated by weight loss measurements. The results of the gravimetric determinations of corrosion rate of aluminium in the presence of blank acid and different concentration of additive solutions and as a function of exposure time period are given in Table 1.

TABLE 1.
Calculated values of corrosion rates of aluminium in 0.5 M HCl in the absence and presence of xanthan gum.

Days	System					
	Blank	1 g/l	2 g/l	3 g/l	4 g/l	5 g/l
1	24.499	23.018	21.878	16.961	15.654	12.605
2	13.338	12.057	11.802	9.275	9.566	7.399
3	9.437	8.586	8.252	6.360	6.571	5.481
4	7.350	6.851	6.333	5.168	5.073	4.247
5	6.206	5.590	5.182	4.346	4.638	3.617

These results show that the corrosion rate of aluminium in 0.5 M HCl decreased with increasing concentration of xanthan gum in inhibited solutions compared with the uninhibited solution. This suggests that the inhibiting action is concentration-dependent. As a function of time, the corrosion rate of aluminium in the test solutions decreased with increase in time of exposure. The corrosion rates for aluminium plotted against time and different concentrations of the additive in 0.5 M HCl solution are shown Fig. 2. Inspection of the plots reveals high corrosion rate of aluminium in 0.5 M HCl solution. The plots also illustrate the decreased corrosion rate on introduction of the additive (xanthan gum) into the corrodent, indicating that xanthan gum actually affords corrosion inhibition of aluminium in the hydrochloric acid solution.

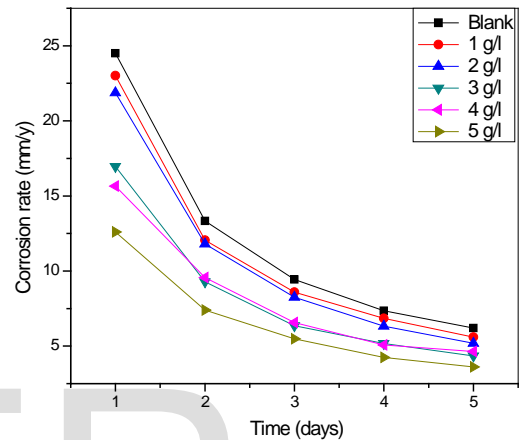


Fig. 2. Plots of corrosion rate of aluminium in 0.5 M HCl in the absence and presence of xanthan gum.

3.2 Inhibition efficiency

The inhibiting action of organic substances like xanthan gum during metal corrosion is due to the adsorption ability of their molecules, where the resulting adsorption film isolates the metal surface from the corrosion medium. Consequently, in inhibited solutions, the corrosion rate is indicative

of the number of free corrosion sites remaining after some sites have been effectively blocked by inhibitor adsorption. The values obtained for the inhibition efficiency of xanthan gum calculated from equation (2) are shown in Table 2.

TABLE 2.
Calculated values of inhibition efficiency (IE %) and surface coverage (θ) of xanthan gum on aluminium corrosion in 0.5 M HCl solution.

Days	Inhibition efficiency (degree of surface coverage)				
	1 g/l	2 g/l	3 g/l	4 g/l	5 g/l
1	6.67 (0.07)	15.56 (0.16)	28.89 (0.29)	40.00 (0.40)	48.89 (0.49)
2	10.20 (0.10)	16.33 (0.16)	28.57 (0.29)	32.65 (0.33)	44.90 (0.45)
3	9.62 (0.10)	17.31 (0.17)	30.77 (0.31)	34.62 (0.35)	42.31 (0.42)
4	7.41 (0.07)	18.52 (0.19)	27.78 (0.28)	35.19 (0.35)	42.59 (0.43)
5	10.53 (0.11)	21.05 (0.21)	28.07 (0.28)	29.82 (0.30)	42.11 (0.42)

If it is assumed that corrosion occurs only at the free sites such that the covered sites have negligible corrosion rates, the degree of surface coverage (θ) of the aluminium by xanthan gum can be assessed by comparing the corrosion rates of the aluminium coupons in absence (C_r^{blk}) and presence of the xanthan gum (C_r^{xg}) using the expression

$$\theta = \left(1 - \frac{C_r^{xg}}{C_r^{blk}} \right) \quad (3)$$

Table 2 shows that values of degree of surface coverage calculated for the various inhibited solutions increased with increasing concentration of xanthan gum. From Fig. 3, inhibition efficiency is observed to increase with increasing concentration of xanthan gum, resulting from enhanced adsorption. As

a function of time, inhibition efficiency did not show significant decrease with time. This implies that the adsorbed inhibitor film is stabilized and is durable under the prevailing experimental conditions. This trend could be explained on the basis of macromolecular nature of xanthan gum whose effect is pronounced at increased concentration.

Owing to the chemical composition of xanthan gum,

it is quite difficult to assign the inhibitive effect to a particular functional group. However, some literatures [31-33] have reported that corrosion inhibition by organic substances is attributed to the presence of oxygen, π electrons, aromaticity etc. Further investigation using surface analytical techniques will enable characterization of the active materials in the adsorbed layer and identification of the most active ingredients.

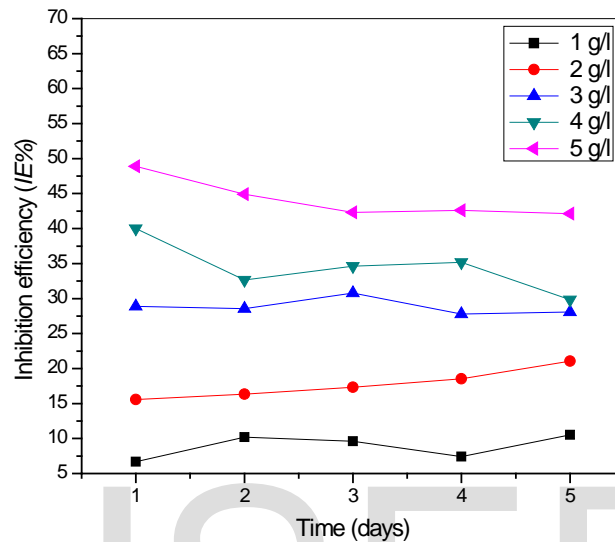


Fig. 3. Plots of inhibition efficiency against time for different concentrations of xanthan gum.

3.3 Adsorption considerations

To gain insight into the nature of adsorption, classical adsorption isotherm of Temkin has been used to represent the adsorption behaviour of xanthan gum on aluminium surface. Temkin isotherm is represented by the expression

$$\exp(-2a\theta) = K_{ads} C$$

(4)

where a is molecules' interaction parameter, θ is the degree of surface coverage, C is the concentration of xanthan gum. Vital information on the nature of interaction between the xanthan gum studied and the corroding aluminium surface has been extrapolated from Figs. 4 and 5 and data presented in Tables 3 and 4.

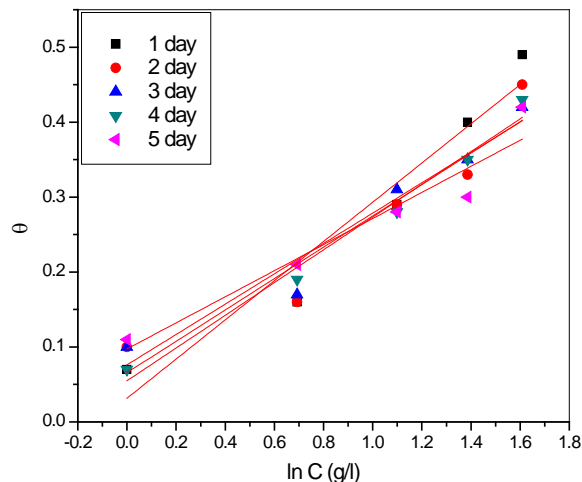


Fig. 4. Temkin adsorption isotherm plots for aluminium corrosion in 0.5 M HCl in the presence of different concentrations of

xanthan gum at ambient temperature, 28 °C.

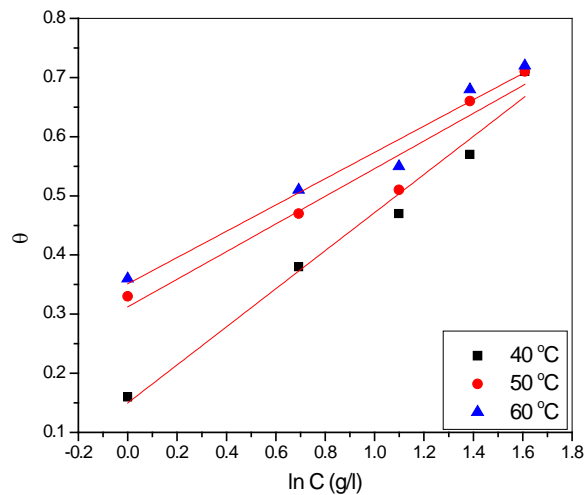


Fig. 5. Temkin adsorption isotherm plots for aluminium corrosion in 0.5 M HCl in the presence of different concentrations of xanthan gum at varying temperatures.

The negative values of a correspond to repulsion and positive values to attraction. Adherence to the Temkin isotherm is further TABLE 3.

Temkin adsorption parameters and associated standard free energy of adsorption for aluminium corrosion in 0.5 M HCl solution using xanthan gum as inhibitor at ambient temperature (28 °C)

Day	a	K_{ads}	ΔG_{ads} (kJ/mol)
1	-1.911	1.128	-10.352
2	-2.391	1.370	-10.839
3	-2.450	1.453	-10.986
4	-2.290	1.286	-10.681
5	-2.873	1.750	-11.452

K_{ads} is the equilibrium constant of adsorption process which is evaluated from the intercepts of the plots and is related to the standard free energy of adsorption given as

$$K_{ads} = \frac{1}{55.5} \exp\left(-\frac{\Delta G_{ads}}{RT}\right) \quad (5)$$

where R and T are universal gas constant and absolute temperature respectively. The Temkin adsorption isotherm plots give straight lines which describe the relationship between the degree of surface coverage (θ) and concentration of inhibitor C .

From the surface coverage data of xanthan gum, it is obvious that the adsorption of the xanthan gum on the aluminium surface inhibits corrosion. Generally, four types of adsorption may take place, involving organic molecules at the metal/solution interface [34]:

- (i) The electrolytic attraction between charged molecules and the charged metal
- (ii) Interaction of unshared electron pairs in the molecules with the metal
- (iii) Interaction of π electrons with metal and

ther evidence of the adsorption of neutral xanthan gum molecules on the aluminium surface from the acid solution.

TABLE 4. Temkin adsorption parameters and associated standard free energy of adsorption for aluminium corrosion in 0.5 M HCl solution using xanthan gum as inhibitor at varying temperatures

Temperature (°C)	a	K_{ads}	ΔG_{ads} (kJ/mol)
40	-1.553	1.593	-11.663
50	-2.139	3.802	-14.372
60	-2.249	4.853	-15.493

- (iv) Combination of the above three points.

Consequently, inhibition occurs and inhibition efficiency depends on several factors such as the number of adsorption sites and their charge density, molecular size, heat of hydrogenation, mode of interaction with the metal surface and the formation of metallic complexes.

The values of standard free energy of adsorption, ΔG_{ads} obtained indicate physical adsorption of xanthan gum on the aluminium surface. However, as a function of temperature, ΔG_{ads} becomes more negative with increase in temperature, suggesting that though adsorption of xanthan gum show physical adsorption, the inhibitor has propensity towards chemisorption behaviour as evidenced in the increase in inhibition efficiency with rise in temperature.

3.4 Effect of temperature

The effect of temperature on the corrosion behaviour of aluminium in uninhibited and inhibited solutions containing different concentrations of xanthan gum was investigated at 40, 50 and 60 °C. The data in Table 5 indicate that the rates of

corrosion in absence and presence of the xanthan gum usually accelerates corrosion processes. increased with rise in temperature since temperature elevation

TABLE 5.

Calculated values of corrosion rate (mm/y) and inhibition efficiency (IE %) for aluminium dissolution in 0.5 M HCl solution in the absence and presence of xanthan gum.

System	Corrosion rate (mm/y)			Inhibition efficiency (IE %)		
	40 °C	50 °C	60 °C	40 °C	50 °C	60 °C
Blank	100.435	125.543	146.467	-	-	-
1 g/l	84.460	84.460	93.350	15.91	32.72	36.27
2 g/l	62.772	66.956	71.141	37.50	46.67	51.43
3 g/l	52.985	61.816	66.232	47.24	50.76	54.78
4 g/l	42.970	42.970	47.267	57.22	65.77	67.73
5 g/l	29.047	36.062	40.570	71.08	71.28	72.30

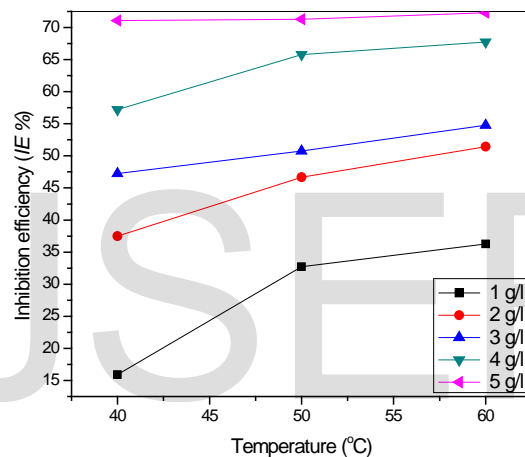


Fig. 6. Plots of inhibition efficiency against temperature for aluminium corrosion in 0.5 M HCl solution in the presence of different concentrations of xanthan gum.

Inhibition efficiency is observed from Fig.6 to increase with rise in temperature. Adsorption of an organic inhibitor can affect the corrosion rate by either decreasing the available reaction area (geometric blocking effect) or by modifying the activation energy of the anodic or cathodic reactions occurring in the inhibitor - free surface in the course of the inhibited corrosion process [35]. The apparent activation energies (E_a)

for the corrosion process in absence and presence of inhibitor were evaluated from Arrhenius equation:

$$C_r = A \exp\left(-\frac{E_a}{RT}\right) \tag{6}$$

where A is the pre-exponential factor and other terms retain their previous meaning.

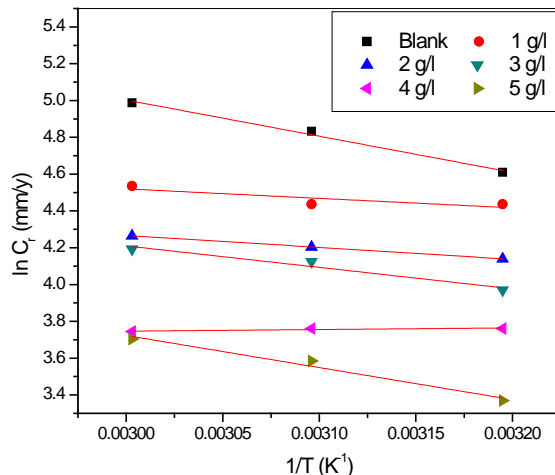


Fig. 7. Arrhenius plots for aluminium dissolution in 0.5 M HCl solution in the absence and presence of different concentrations of xanthan gum.

The variation of logarithm of corrosion rate with reciprocal of absolute temperature is shown in Fig.7. The calculated values of E_a are given in Table 6. Addition of xanthan gum is seen to lower the apparent activation energy, E_a for the corrosion reaction. The observed trend can be explained on the basis that increase in inhibition efficiency with rise in temperature including lowering of E_a in the presence of xanthan gum

suggests likely specific interaction between xanthan gum and the aluminium surface. Such behaviour has been attributed to a modification in the nature of adsorption, whereby the inhibitor is physically adsorbed at low temperature, whereas chemisorptions are favoured at higher temperatures. Similar interpretations can be found in the literature [36, 37].

TABLE 6.

Calculated values of activation energy E_a , heat of adsorption Q_{ads} , enthalpy ΔH° and entropy ΔS° of activation for aluminium corrosion in 0.5 M HCl solution in the absence and presence of xanthan gum

System	E_a (kJ/mol)	Q_{ads} (kJ/mol)	ΔH°	ΔS°
Blank	16.351	-	12.229	-197.379
1 g/l	4.284	16.717	-17.405	-197.480
2 g/l	5.412	19.910	-8.567	-197.460
3 g/l	9.692	13.659	-4.193	-197.448
4 g/l	-0.728	33.125	-34.630	-197.551
5 g/l	14.502	6.533	13.122	-197.409

An estimate of the heats of adsorption (Q_{ads}) was evaluated from the kinetic thermodynamic model [38]

$$\left(\frac{\theta}{1-\theta}\right) = A.C \exp\left(\frac{-Q_{ads}}{RT}\right) \tag{7}$$

where A is a constant, C is the concentration of inhibitor, θ is the occupied and $(1-\theta)$ is the vacant site not occupied by inhibitor. Plots of $\ln(\theta/1-\theta)$ against $1/T$ are shown in Fig.8. The plots also reveal the different characters of adsorption of xanthan gum.

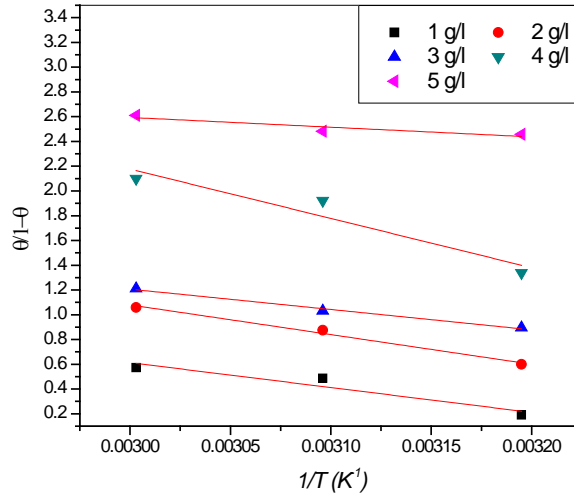


Fig.8: Kinetic-thermodynamic plots for aluminium dissolution in 0.5 M HCl solution in the presence of different concentrations of xanthan gum.

The calculated values of Q_{ads} are given in Table 6. The positive values indicate that inhibitor adsorption and hence inhibition increases with rise in temperature, while negative values mean the opposite effect [39]. Some other activation parameters such as the enthalpy change of activation (ΔH^o) and entropy change of activation (ΔS^o) were obtained from the Eyring transition equation

$$\ln\left(\frac{C_r}{T}\right) = -\left(\frac{\Delta H^o}{RT}\right) + \ln\left(\frac{R}{N_A h}\right) + \left(\frac{\Delta S^o}{R}\right) \quad (8)$$

where N_A is the Avogadro number, h is Planck's constant and other terms also retain their previous meanings.

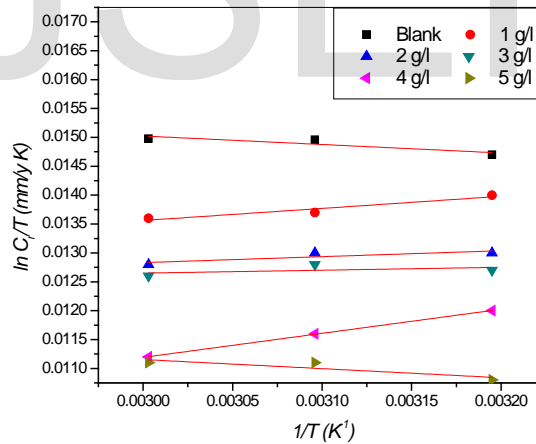


Fig. 9: Eyring transition plots for aluminium dissolution in 0.5 M HCl solution in the absence and presence of different concentrations of xanthan gum.

Fig.9 shows plots of $\ln (C_r/T)$ against $1/T$ with and without xanthan gum and gives straight lines with slope ($-\Delta H^o/R$) and intercept [$\ln (R/N_A h) + (\Delta S^o/R)$]. The calculated values of ΔH^o and ΔS^o obtained are given in Table 6. The positive values of ΔH^o both in absence and presence of xanthan gum reflect the endothermic nature of the aluminium dissolution process whereas the negative values imply exothermic

process. It is also clear that the activation enthalpies vary in the same manner as the activation energies, supporting the inhibition mechanisms. Large and negative values of entropies imply that the activated complex in the rate determining step represents an association rather than a dissociation step, meaning that a decrease in disordering takes place on going from reactants to the activated complex.

3.5 Potentiodynamic polarization results

Polarization experiments were conducted with uninhibited 0.5 M HCl and 5.0 g/l inhibited test solutions to gain insight on the effect of xanthan gum on the kinetics of anodic and cathodic partial reactions occurring on the aluminium electrode in uninhibited and inhibited 0.5 M HCl solutions. Fig.10 illustrates the cathodic and anodic polarization curves recorded for aluminium corrosion in 0.5 M HCl in the absence and presence of xanthan gum. The anodic and cathodic branches of the polarization curves display behaviour within the studied potential range. The values of the corrosion current density were determined by extrapolation of the cathodic and anodic lines to the corrosion potential. It follows from Fig.10 that the cathodic partial hydrogen evolution reaction was markedly affected by the inhibitor, while the anodic one is slightly affected. The effect of anodic partial inhibitive reaction diminished with increasing corrosion potential whereby the effect is eliminated at corrosion potential 0.25 V. Based on the fact that E_{corr} is not altered to any significant extent, the implication is that the corrosion inhibition process is under mixed control with predominant cathodic effect. Therefore, the corrosion inhibition effect of xanthan gum is contributed significantly by cathodic partial inhibition reaction and also by slight reduction of anodic partial reaction. However, increase in corrosion potential, at an extent, exerted a stimulating effect on the anodic partial reaction, suggesting that the anodic dissolution reaction is the predominant reaction determining the corrosion

rate of the system. The corrosion potential for uninhibited and inhibited systems are -360.44 and -327.46 mV/SCE respectively while the corrosion current densities are 5031.6 and 71878 $\mu\text{A cm}^{-2}$ for uninhibited and inhibited systems respectively.

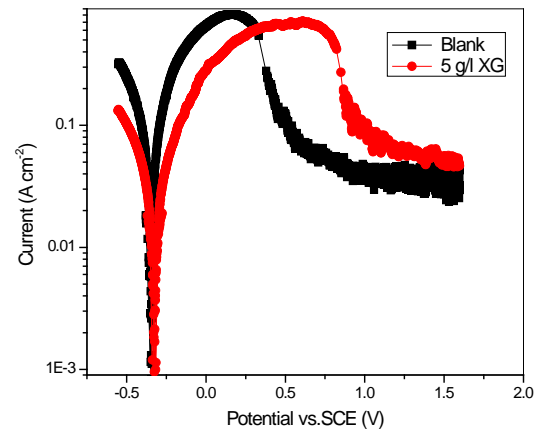
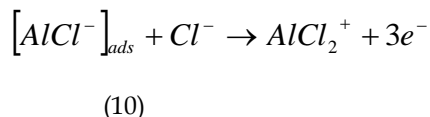


Figure 10: Polarization curves for aluminium corrosion in 0.5 M HCl in the absence and presence of Xanthan gum.

3.6 Mechanism of inhibition

Adsorption of xanthan gum on the aluminum surface is followed by replacement of pre-adsorbed water molecules and modification of the low stable aluminium surface in the acid medium to a more stable aluminium-inhibitor complex. The more stable complex formed directly on the basis of donor - acceptor interactions between π electrons, oxygen atoms and the carboxylic functions of the neutral species as well as the π electrons of cationic species and the vacant d orbitals of aluminium. It has also been reported that the adsorption of the heterocyclic compounds occurs with the aromatic rings sometimes parallel but mostly normal to the aluminium surface [33, 40].

However, in acid solutions, the majority of organic compounds do exist as protonated species and others in the molecular form. The mechanism of adsorption of protonated xanthan gum on aluminium can follow the proposed reaction sequence of aluminium corrosion in hydrochloric acid reported by Pinto *et al.*, [41]:



It is an acceptable fact that chloride ions have a small degree of hydration, and due to specific adsorption, they should be first adsorbed on the positively charged metal surface. The adsorption of chloride ions creates an excess negative charge towards

the solution side of metal and favour more adsorption of cations [41-43]. Then, the inhibitor molecules adsorb through electrostatic interactions between the negatively charged metal surface and positively charged inhibitor molecule xanthan gum and form a protective metal-inhibitor complex adsorption layer. By this, the oxidation reaction can be prevented. In a similar manner, protonated xanthan gum can electrostatically interact with $AlCl^-_{ads}$ species, and then the oxidation of $AlCl^-_{ads}$ to $AlCl_2^+$ can be prevented as well.

Conclusion

Xanthan gum has been found to be an inhibitor for aluminium corrosion in 0.5 M HCl solution and its inhibition efficiency increases with increasing concentration. The corrosion process is inhibited by the adsorption of xanthan gum on the aluminium surface following the Temkin isotherm. The inhibiting action is attributed to general adsorption of protonated and molecular species on cathodic and anodic sites on the corroding metal surface. Inhibition efficiency increases with rise in temperature and addition of xanthan gum leads to a decrease in apparent activation energy. The activation enthalpy, ΔH^0 and activation entropy, ΔS^0 as well as heat of adsorption, Q_{ads} were calculated and corroborates the proposed inhibition mechanisms. Polarization studies suggest that the corrosion inhibitive effect of xanthan gum is contributed substantially by cathodic partial inhibitive reaction whereas the

anodic dissolution reaction is the predominant reaction de-

termining the corrosion rate of the system.

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