EFFECT OF ADDITION OF THE BIO MOLECULE NICOTINIC ACID IN THE CORROSION OF ALUMINIUM IN HYDROCHLORIC ACID SOLUTION

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ABSTRACT: Corrosion control of metals is of technical, economical, environmental, and aesthetical importance. The use of corrosion inhibitors constitutes one of the most economical ways to mitigate the corrosion rate, protect metal surfaces against corrosion and preserve industrial equipments. The environmental toxicity of organic corrosion inhibitors has prompted the search for green corrosion inhibitors as they are biodegradable, do not contain heavy metals or other toxic compounds. Nicotinic acid-Pyridine-3-carboxylic acid is a water-soluble vitamin belonging to the vitamin B family that is widely distributed in foods such as milk, wheat germ, and meat, and can be synthesized in the body from tryptophan. The present study is concerned with the inhibition effect of easily biodegradable and environment friendly nicotinic acid on the corrosion of Aluminium in HCl. The corrosion of Aluminium in the presence of *nicotinic acid* in 0.3M HCl is studied using the weight loss method at 303, 308, 313 and 318K.The results obtained reveals that corrosion of Aluminium by HCl is inhibited in the presence of Nicotinic acid. The inhibition efficiency increases with increasing inhibitor concentration and at the same time inhibition efficiency decreases with increasing temperature. Inhibition occurs through adsorption of nicotinic acid on the metal surface. Langmuir adsorption isotherm is obeyed. The corrosion inhibition is also confirmed by Tafel polarization study, Impedance spectroscopy and SEM analysis.

Keywords: Nicotinic acid, aluminium, Tafel plot, impedance

1. INTRODUCTION

Aluminum is an important metal in industry owing to its many excellent characteristics including its lightweight, strength, durability, formability, high thermal and electrical conductivity, and good corrosion resistance. It is widely used as a material for automobiles, aviation, household appliances, containers, reaction vessels, pipes, machinery and chemical batteries. Aluminium forms a protective layer of oxide on its surface but this oxide dissolves and makes the metal susceptible to corrosion in acid, salty and alkaline medium [1,2,3]. Acid media are always used in the study of corrosion of aluminum because acids (HCl, H_2SO_4, H_2PO_4, CH_3COOH, etc) are commonly used for pickling, industrial cleaning, descaling, chemical and electrochemical etching of aluminium etc, [4,5,6].

The use of inhibitors for the control of corrosion of materials, which are in contact with aggressive environment, is an accepted practice. Corrosion inhibitors are substances, which when added in small concentrations to corrosive media decrease or prevent the reaction of the metal with the media. Corrosion protection by inhibitors has been employed in many systems namely; cooling system, refinery units, chemicals, oil and gas production units' boiler, etc. [7-11_]. Using inhibitors is an effective method to reduce corrosion rate [12]. Chemical inhibitors play an important role in the protection and mitigation strategies for retarding corrosion [13]. Corrosion inhibitors are generally organic compounds containing N, O, S heteroatoms or structures containing π electrons in their molecules [14]. Inhibitors decrease the corrosion rate by adsorbing on the metal surface and blocking the active sites by displacing water molecules and form a compact barrier film on the metal surface [15]. Presence of electron releasing group or electron attracting group in the pyridine ring increases or decreases the availability of electrons on the nitrogen atom of the pyridine ring. If electrons are freely available on nitrogen atom, then, adsorption of the inhibitor on the metal surface increases and hence inhibition efficiency increases. Otherwise non-availability of electrons on the nitrogen atom decreases the efficiency of adsorption on the metal atom and hence the inhibition efficiency decreases [16]

2. EXPERIMENTAL

2.1 Materials and Methods

Aluminium samples having an elemental composition of Al- 99.3, Si-0.240%, Fe-0.241%, Cu-0.035%, Mn-0.102%, Ti-0.019%, Pb-0.014%, Zn- 0.043% are used. The samples are cut to dimensions of 4 cm×1

cm×0.15 cm, and the surface is abraded with different emery papers 400, 600 and 1000 grades and washed with acetone. The cleaned samples are then washed with doubly distilled water and finally dried.

2.2 Preparation of Stock Solution

1 gm of nicotinic acid is dissolved in 0.3M HCl, and then made upto 100 ml in a standard measuring flask. 0.2, 0.4, 0.6, 0.8 and 1ml of this solution is diluted to 100 ml, which yields exactly 0.0001N, 0.0003N, 0.0004N, 0.0006N, 0.0008N of nicotinic acid solution which is used for the analysis.

2.3 Determination of Surface Area of the specimens

The length, breadth and the thickness of aluminium specimens and the radius of the holes are determined with the help of vernier calipers of high precision and the surface areas of the specimens are calculated.

2.4 Weighing the specimens before and after corrosion

The weights of the specimens before and after immersion are determined using a balance Shimadzu AY62 model.

2.5 Weight loss method

The weighed specimen, are suspended by means of glass hooks in 100 ml beakers containing 100 ml of aqueous solution of 0.3M HCl containing various concentrations of the inhibitor. After 1 hour immersion the specimens are taken out, washed in running water, dried and weighed. From the change in weights of the specimen, corrosion rates are calculated using the following relationship.

$$\text{Corrosion rate} = \frac{[\text{loss in weight (mg)}]}{[\text{Surface area of the specimen (dm}^2) \times \text{period of immersion (hours)}]} \quad ----(1)$$

ϱ-density of the metal in g/cm²

The percentage of inhibition efficiency was calculated using the following equation

$$\text{IE\%} = \frac{CR_0 - CR_t}{CR_0} \times 100 \quad ------------ (2)$$

CR_0 = corrosion rate in the absence of inhibitor and

CR_t = corrosion rate in the presence of inhibitor

2.6 Temperatures Effects

Weight loss measurements are performed in 0.3M HCl with various concentration of inhibitor at various temperature are calculated. Each experiment is duplicated to get good reproducibility.

2.7 Adsorption isotherm

The adsorption isotherm can be determined by assuming that inhibition effect is mainly due to the adsorption at metal /solution interface. Basic information on the adsorption of inhibitor on the metal surface can be provided by adsorption isotherm. In order to obtain the isotherm the fractional surface coverage values (θ) as a function of inhibitor concentration must be obtained. The values of θ can be easily determined from the weight loss measurements by the ratio. % I.E /100, where % I.E is inhibition efficiency is obtained by weight loss method. So it is necessary to determine empirically which isotherm fits best to the adsorption of inhibitor on the Aluminium surface.

2.8 Polarization measurements

The working electrode is immersed in test solution during 30 minutes until a steady state open circuit potential is obtained. Both anodic and cathodic polarization curves are recorded by potentiodynamically (lm using a corrosion measurement system consisting of a BAS Model 100 KHZ to 10MHZ a comprised elctrochemical analyzer (made in Lafeyette, in USA) PL-10 digital plotter (DMP -40 series the instruments, Division, Hovston, TX, USA) made by a platinum for and Hg/ Hg$_2$Cl$_2$ / 0.3M HCl are used as auxiliary electrode and reference electrode respectively. This is controlled by a personal computer.

2.9 EIS measurements

The electro chemical impedance spectroscopy is carried out using a transfer function analyzer, with a small amplitude ac. The double layer capacitance (C_{dl}) and the charge transfer resistance (R_{ct}) are obtained using AC impedance instrument Princeton Applied Research (Model – 7310).

2.10 Surface analysis

The aluminum specimens are immersed in 0.3 M HCl for an immersion time of 1 h in the absence and presence of optimum concentration of NA. After 1 h, the specimens are taken out and their surface morphology is determined using scanning electron microscopy (SEM) technique. The presence of surface elements are determined using high energy disperses X-ray (EDX) technique. The SEM and EDS analysis are performed using BRUKER VEGA 3 TESCAN instrument at 500 magnification.

3. RESULTS AND DISCUSSION

Molecular structure of organic compound used in the present study is given in Fig.

**Fig. 1.** Structure of Nicotinic acid
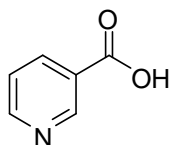
3.1 Weight loss measurements

The corrosion rate (CR) obtained from the weight loss method at different concentrations of NA alone and NA with KI in 0.3M HCl at various temperatures are summarized in Table 1. The inhibition efficiency and the surface coverage value θ are calculated and given in Table1. The variation of inhibition efficiency with increase in inhibitor concentrations of NA with KI (B) and without KI (A) at various temperatures   are shown in Fig 2. and the variation of corrosion rate with increase in concentration of NA with KI (B) and without KI (A) at various temperatures are given in fig.3.
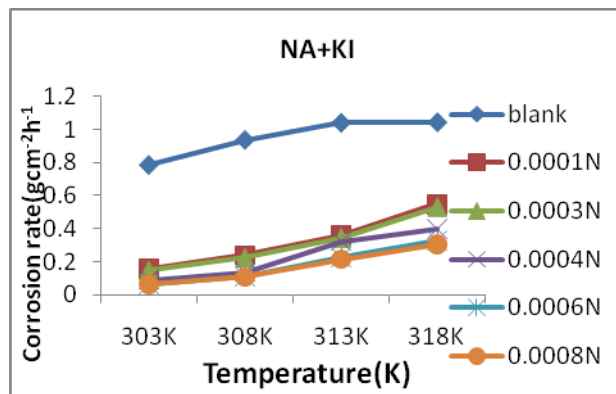
Table 1. Corrosion of **Aluminium** in **0.3M HCl** in the presence and absence of Various **concentrations** of **Nicotinic acid** with and without KI at various **Temperatures**

| NA (N)	Corrosion Rate$\times 10^{-2} g cm^{-2} h^{-1}$				Inhibition Efficiency （%）				Θ			
	303 K	308 K	303K	308K	313K	318K	313K	318K	303K	308K	313K	318K
Blank	0.7861	0.9359	-	-	-	-	-	-	-	-	-	-
0.0001	0.2299	0.3754	0.7075	0.5988	0.4476	0.3488	44.76	34.88	0.7075	0.5988	0.4476	0.3488
0.0003	0.1872	0.3662	0.7618	0.6087	0.4857	0.3888	48.57	38.88	0.7618	0.6087	0.4857	0.3888
0.0004	0.1645	0.2977	0.7907	0.6819	0.5044	0.3995	50.44	39.95	0.7907	0.6819	0.5044	0.3995
0.0006	0.1456	0.2793	0.8147	0.7015	0.5533	0.4445	55.33	44.45	0.8147	0.7015	0.5533	0.4445
0.0008	0.1410	0.2476	0.8206	0.7354	0.5698	0.4759	56.98	47.59	0.8206	0.7354	0.5698	0.4759
NA +KI												
Blank	0.7861	0.9359	1.0364	1.0417	-	-	-	-	-	-	-	-
0.0001	0.1570	0.2391	0.3593	0.5497	80.02	74.45	65.33	47.23	0.8002	0.7445	0.6533	0.4723
0.0003	0.1463	0.2234	0.3452	0.5326	81.38	76.12	66.69	48.87	0.8138	0.7612	0.6669	0.4887
0.0004	0.0905	0.1356	0.3217	0.3964	88.48	85.51	98.95	61.94	0.8848	0.8551	0.9895	0.6194
0.0006	0.0648	0.1146	0.2292	0.3306	91.75	87.75	77.88	68.26	0.9175	0.8775	0.7788	0.6826
0.0008	0.0601	0.1046	0.2136	0.3044	92.35	88.82	79.39	70.77	0.9235	0.8882	0.7939	0.7077

Fig. 2 Variation of inhibition efficiency on Aluminium corrosion with different concentration of NA with and without KI in 0.3M HCl at 303K, 308K, 313K and 318K for 1 hour
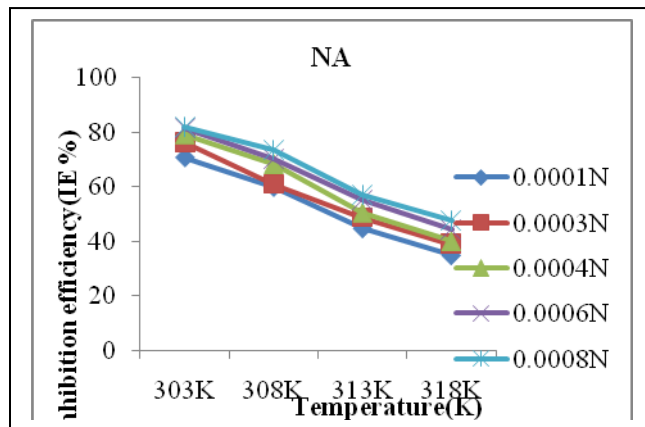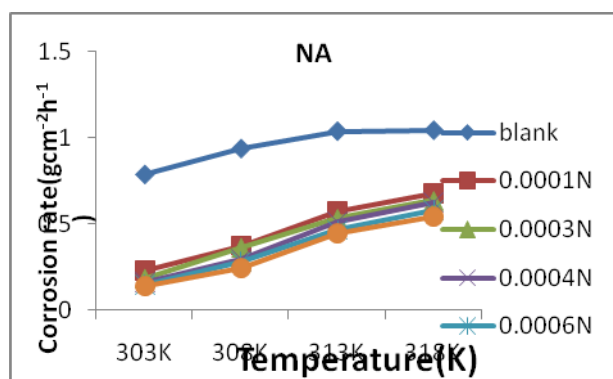

A

B
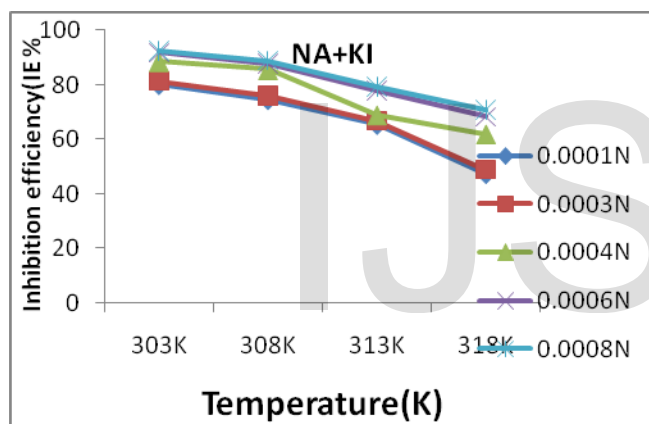
B

Fig. 3. Variation of corrosion rate on Aluminium corrosion as a function of temperature for NA with and without KI in 0.3M HCl at 303K, 308K, 313K and 318K for 1 hour

A

It is observed that NA inhibits the corrosion of Aluminium in HCl solution at all concentrations used in the study. At the same time, when KI is used along with NA, the maximum inhibition efficiencies are obtained. A synergistic effect is noticed between NA and I$^-$ ions. The corrosion inhibition increases, with increase in inhibitor concentration (Fig. 2). and hence the corrosion rate decreases. This trend may result from the fact that adsorption and surface coverage increases with the increase in concentration.

The CR increases with increasing the temperature as shown in Fig 3. This indicates that the augmentation of temperature led to the reduction of the inhibitor adsorption and acceleration of the dissolution process.
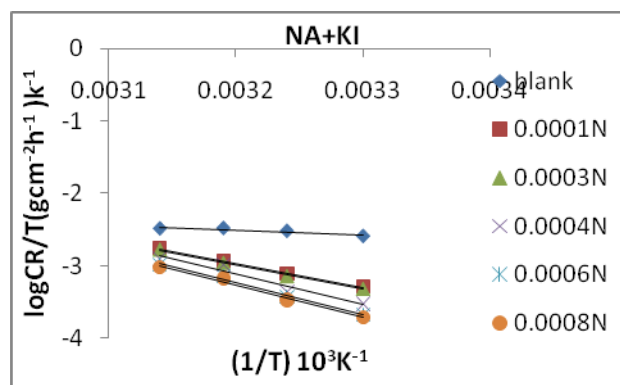
According to Arrhenius equation

$$\text{Log (CR)} = \frac{-Ea}{2.303RT} + log\lambda \quad \text{----------- (3)}$$

Where Ea is the apparent effective activation energy, R is the molar gas constant and λ is the Arrhenius pre exponential factor. The plot of log CR vs 1/T based on Arrhenius equation is shown in fig 4
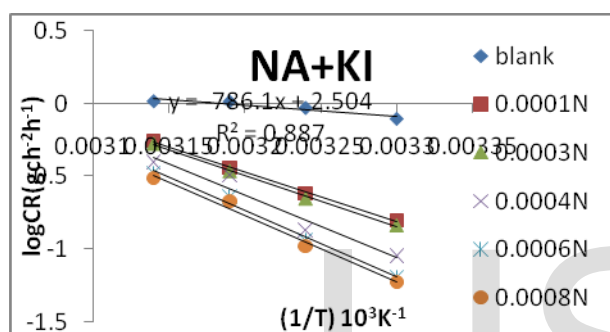
Fig 4 Arrhemius plots for aluminium corrosion in 0.3M HCl different concentration of NA with (B) and without KI (A) .
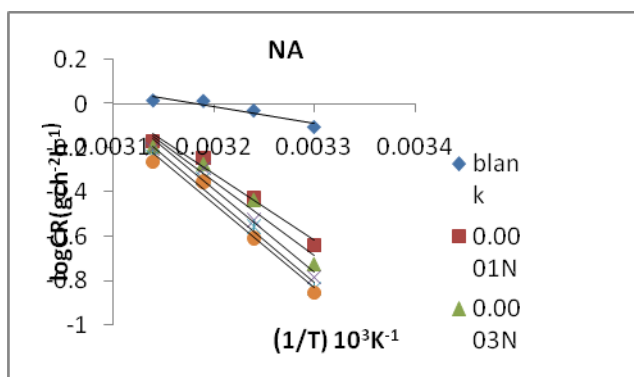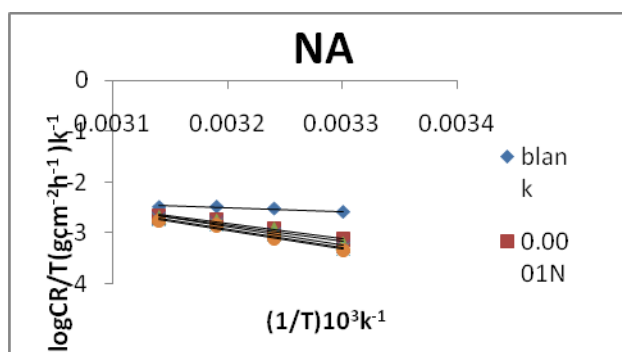
A

B

B

According to transition state theory

$$CR = \frac{RT}{Nh} \exp\left(\frac{\Delta S^*}{R}\right) \exp\left(\frac{-\Delta H^* ads}{RT}\right) \text{————— (4)}$$

where h is Planck's constant, N is Avogadro's number, ΔH* is the activation enthalpy and ΔS* is the activation entropy. The plot of LogCR/T against 1/T in accordance with ARRT is shown in fig5

Fig. 5. Transition state plot for aluminium corrosion in 0.3M HCl in different concentration of NA with (B) and without KI (A)

A

The calculated values of the apparent activation energy, Ea *, from the plot of Arrhenius equation and activation enthalpies, ΔH* and activation entropies, ΔS* from the plot of ARRT are given in Table 2.

Table 2. Activation parameter and thermodynamic parameters for aluminium corrosion in 0.3M HCl in various concentrations of NA with and without KI.

NA (N)	E_a (KJ mol^{-1})		ΔH^* (KJ mol^{-1})		ΔS^* (J $mol^{-1}K^{-1}$)	
	NA	NA+KI	NA	NA+KI	NA	NA+KI
Blank	15.051		12.541		-1.086	
0.0001	57.84	65.23	55.31	62.70	-0.95	-0.93
0.0003	64.10	67.47	61.59	64.94	-0.93	-0.92
0.0004	71.76	82.84	69.23	80.32	-0.91	-0.88
0.0006	73.29	87.55	70.76	85.03	-0.90	-0.86

These values indicate that the presence of the additives increase the activation energy, Ea * and the activation enthalpy, ΔH* and decrease the activation entropy ΔS* for the corrosion process. The addition of inhibitors modify the values of Ea*. This is may be attributed to the adsorption of inhibitors on Al surface and this adsorption make an energy barrier

and this energy barrier of corrosion reaction increases as the concentration of the inhibitor is increased, and hence % IE increases. This suggested that the process is activation controlled. The entropy of activation (ΔS^*) in the blank and inhibited solutions is large and negative indicating that the activated complex represents association rather than dissociation step. Addition of KI to the inhibitor further increases the energy of activation and the enthalpy of activation and thus make the dissolution process more difficult and hence corrosion rate is diminished to a greater extent and inhibition efficiency increases and a maximum inhibition efficiency of 92%is achieved at room temperature. However at higher temperature the inhibition efficiency decreases for the inhibitors with and without KI . This may be due to the acceleration of desorption process at higher temperatures.

3.3 Adsorption isotherm

The surface coverage values (θ)from the table 1 are fitted into the Langmuir adsorption isotherm. The Langmuir adsorption isotherm model has the form $C/\theta = 1/K_{ads} + C$. Where C is the concentration, K_{ads} is the equilibrium Constant of adsorption process. A plot of C/θ Vs C is linear and is shown in Fig.6 The regression coefficient and the slope of the lines are noted and tabulated in Table3.

Fig. 6. Langmuir adsorption isotherm plot for corrosionof aluminium in 0.3 M HCl containing different concentration of NA with (A) and without KI (B) at different temperatures
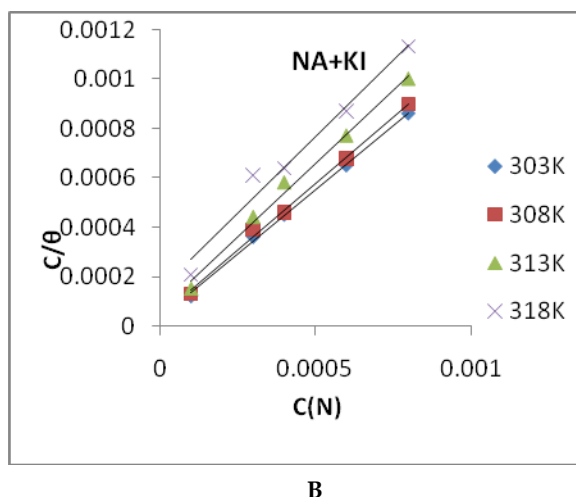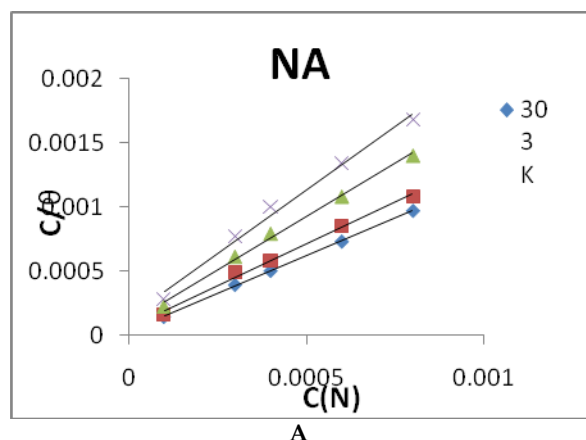
B

Table 3. Adsorption parameters calculated from the Langmuir adsorption isotherm for NA and NA +KI

Inhibitor	R²		Slope	intercept	$K_{ads}M^{-1}$	$\Delta G^{\circ\circ}_{ads}$ KJ mol^{-1}
NA	303	0.999	1.177	0.00003	33333	-36.35
	308	0.993	1.296	0.00006	16666	-35.18
	313	0.995	1.661	0.00009	11111	-34.69
	318	0.990	1.973	0.00025	4000	-32.55
NA +KI	303	0.998	1.042	0.000026	38461	-36.71
	308	0.996	1.080	0.00004	25000	-36.22
	313	0.991	1.180	0.00006	16666	-35.75
	318	0.973	1.245	0.00024	4166	-32.66

From the table it is found that the R² values for both NA and NA+KI are closer to unity, This shows that it obeys Langmuir adsorption isotherm at all temperatures. The slope deviates from unity at higher temperatures for NA. This indicates that there is attraction or repulsion in the adsorbed layer of inhibitor on the aluminium surface at higher temperatures. However for NA+KI the slope remains closer to unity which shows that Langmuir adsorption isotherm is perfectly obeyed. This leads to the conclusion that every site holds only one inhibitor and there is no interaction between the molecules (17).

K_{ads} values are   calculated   from the equation

A

$$K_{ad\,s} = \frac{1}{intercept} \quad \text{-----------------------------(5)}$$

The K_{ads} is related to the standard free energy of adsorption ΔG°_{ads} by the formula

$$K_{ads} = \frac{1}{55.55} \exp\left(\frac{-\Delta G^{\circ}_{ads}}{RT}\right) \quad \text{------------------(6)}$$

ΔG^{0}_{ads} values are calculated using the above equation and tabulated in table 3.
The values of ΔG^{0}_{ads} are negative and range from –36.71 to –32.55 KJ mol^{-1} for both NA and NA+KI .The negative values indicate that the adsorption of the inhibitors on the aluminium surface is spontaneous. The ΔG^{0}_{ads} values decrease with the increase of temperature suggesting that there is a decrease in adsorption of the inhibitors as the temperature increase from 303 to 318K. ΔG^{0} values less than 40KJ is indicative of physical adsorption and greater than 40kJ is indicative of chemical adsorption. Here for both NA and Na+KI ΔG^{0} values are closer to 40KJ which shows that the mode of adsorption are mixed type. . Hence it is suggested that chloride/iodide molecules are first adsorbed on the sites first making the surface negative and the protonated inhibitor are held on the surface by electrostatic interaction.

3.3. Electrochemical measurements
3.3.1. Tafel method

Fig. 7. Potentiodynamic polarization curves for corrosion of aluminium in 0.3 M HCl alone containing NA with KI.

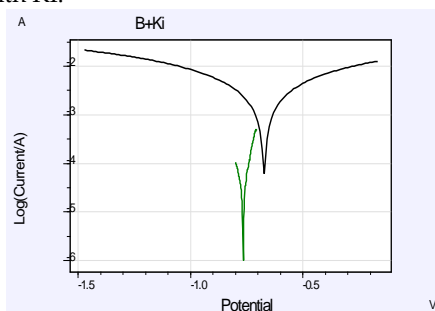
Figure 7 shows typical current-potential of Al in 0.3M HCl in the absence and presence of NA+KI. Electrochemical parameters such as current density (i_{corr}), corrosion potential (E_{corr}), Tafel slopes and as function of inhibitor concentrations in 0.3M HCl are given in Table 4.

Table 4. Corrosion parameters of aluminium immersed in the absence and presence of inhibitor with and without KI obtained by potentiodynamic polarization studies

It is observed from the Tafel curve that addition of

Corrosion parameters	Blank	NA + KI
E_{corr}	-0.6931 (V)	-0.7661 (V)
I_{corr}	0.003218 (A/cm^{2})	0.00002 (A/cm^{2})
R_{P}	38.77 (ohm)	314.3 (ohm)
β_{a}	0.604 (V/dec)	0.039 (V/dec)
β_{c}	0.950 (V/dec)	0.043 (V/dec)
Corr. Rate	35.1 (mm/Y)	0.2274 (mm/Y)

inhibitor system changes both cathodic and anodic reaction which is evident from the change in the anodic and cathodic slope. There is a large reduction in the I_{corr} value which is a measure of the corrosion rate. This confirms the results obtained by weight loss method. The E_{corr} value is shifted towards more negative value on the addition of inhibitor system. This shows that the inhibitor is a cathodic type of inhibitor and controls the cathodic process more predominantly (18, 19).

3.3.2 Electrochemical Impedance spectroscopy-The impedance spectrum of aluminium in 0,3M HCl in the presence and absence of inhibitor system is shown in fig **8**. The results of impedance parameters (R_{ct} and C_{dl}) for inhibitor are given inTable 5.

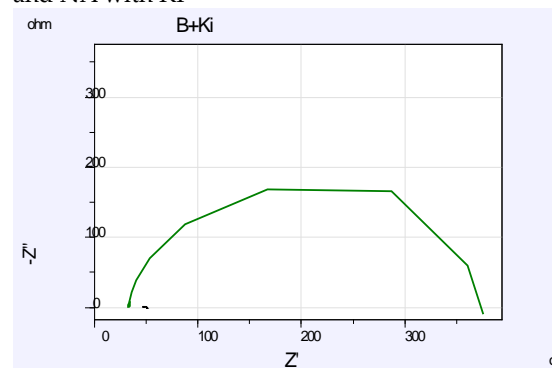**Fig. 8.** Nyquist plots of aluminium in 0.3 M HCl alone and NA with KI

Table 5. Impedance parameters of aluminium in the presence and absence of inhibitor obtained by impedance spectra.

| Impedance parameters	Blank	NA+KI
R_{ct}	3.557 (ohm)	338.7 (ohm)
C_{dl}	1.686×10^{-4} (F)	1.663×10^{-5} (F)
Inhibition efficiency	-	98 %

It can be seen from the table that the charge transfer resistance (R_{ct}) value increase on the addition of inhibitor system NA+KI but the value of double layer capacitance (C_{dl}) decreases due to decrease in local dielectric constant and increase in thickness of the electrical double layer, suggesting that the inhibitor molecules function by adsorption at the metal solution interface. Hence the inhibition efficiency increases (17).

3.4 surface analysis The metal pieces are immersed in 0.3M HCl with and without the inhibitor system for one hour and sem images of the polished surface without immersion and after immersion in 0.3M HCl with and without inhibitors are taken and given in fig 9.
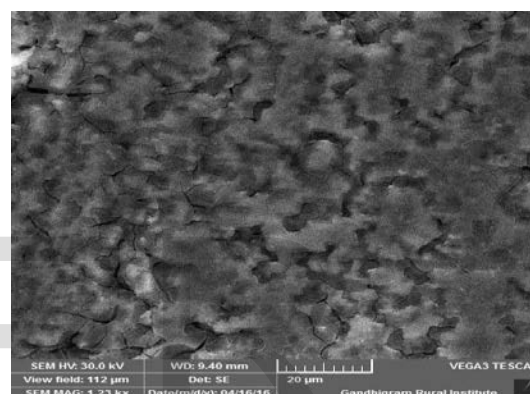
Fig. 9. SEM images of aluminium (a) Polished (b) immersed in 0.3M HCl (c) immersed in 0.3M HCl + NA + KI.


B

C

A
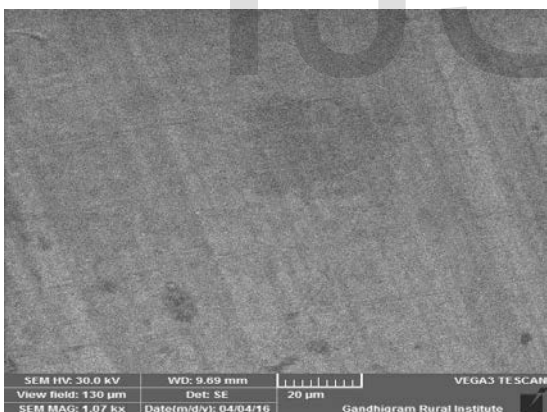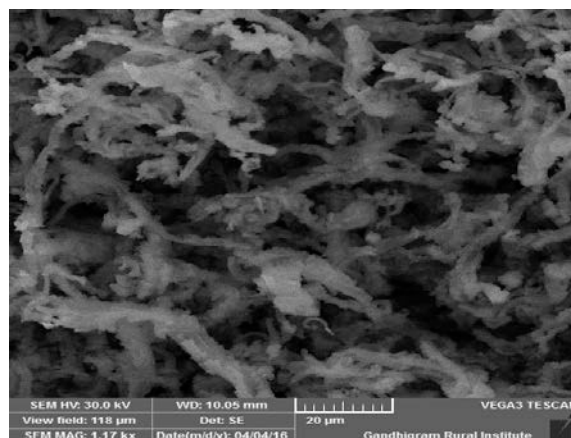
SEM micrographs and the corresponding EDS spectra of the surfaces the samples are recorded in order to see the changes that occurred during the corrosion of aluminium in the presence and absence of the inhibitor. Figure 9 shows that the aluminium immersed in 0.3 M HCl is highly damaged; it can be concluded that the aluminium surface is highly corroded in aggressive acid media. This type of corrosion is typical in aggressive acid solutions. Figure 9 shows a smooth surface with deposited inhibitor on the surface of mild steel after the addition of inhibitor NA+KI to the 0.3M HCl solution. It is clearly seen from the SEM images that the irregularities in the surface due to corrosion are absent on the Aluminium surface and the surface is almost free from corrosion.

The EDS images of the surface of the polished aluminium without immersion, aluminium immersed in 0.3 M HCl and aluminium immersed in 0.3M HCl with inhibitor system for one hour are shown in fig. 10.
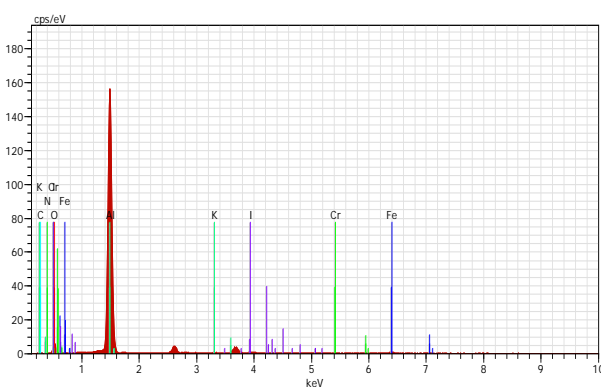
Fig. 10. EDS images of aluminium (A) Polished (B) immersed in 0.3M HCl (C) immersed in 0.3M HCl + NA + KI.
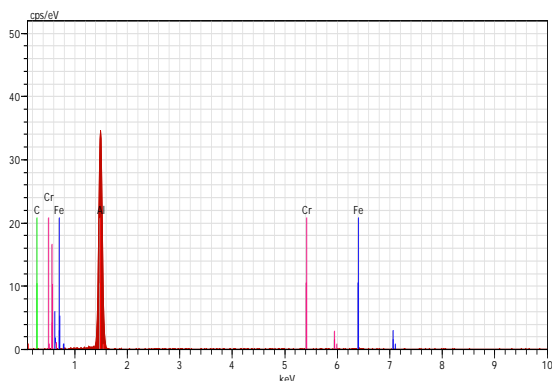
A
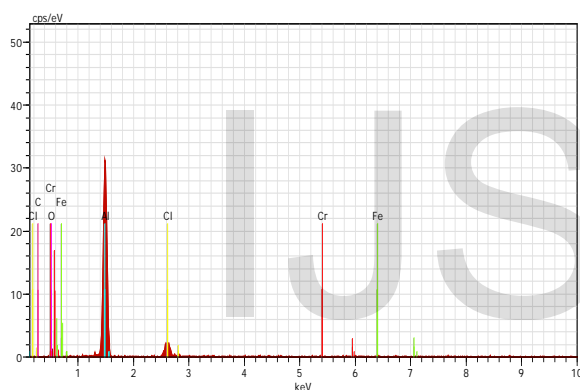
B

C

The EDS data indicate the absence of chloride ion on the metal surface on the inhibited surface (Figure 10). The presence of chloride ion in the blank and the absence of Chloride ion in the presence of inhibitor , the presence of iodide ion on the metal and the presence of nitrogen on the surface

when inhibitor system is added clearly prove that inhibition is due to adsorption of I⁻ and NA. From these results, it can be clearly concluded that NA forms a uniform adsorbed layer on the surface and retards the corrosion.

4. CONCLUSION

Corrosion inhibition in the presence of *nicotinic acid* and KI in 0.3M HCl is studied using the weight loss techniques at 303, 308, 313 and 318K. The results obtained reveal that the inhibition efficiency increases with increasing inhibitor concentration, at the same time inhibition efficiency decreases with increasing temperature. The isotherm reveals that the inhibitor is adsorbed by mixed adsorption. The corrosion inhibition and the formation of adsorbed layer is confirmed by Tafel polarization, Impedance spectroscopy and SEM analysis.

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