ADSORPTION AND THERMODYNAMIC INVESTIGATION OF Al-PURE IN HYDROCHLORIC ACID USING o-AMINOPHENOL-N-SALICYLIDENE AND p-AMINOPHENOL-N-SALICYLIDENE AS CORROSION INHIBITOR

P. M. Wadhwani[1], D. G. Ladha[2], V. K. Panchal[3], N. K. Shah[1*]

[1,2]*Department of Chemistry, School of Sciences, Gujarat University, Ahmedabad-380009, India*
[3]*R.G. Shah Science College, Vasna, Ahmedabad-380009, India*

ABSTRACT

The inhibiting action of a Schiff bases o-Aminophenol-N-Salicylidene and p-Aminophenol-N-Salicylidene derived from salicylaldehyde and substituted aniline towards the corrosion behaviour of aluminium in 1.0 M HCl solution has been studied using weight loss techniques. At constant temperature, the corrosion rate decreases with increasing inhibitor concentration. However, at any inhibitor concentration, the increase in temperature leads to an increase in the corrosion rate of aluminum and decrease in inhibition efficiency. o-APNS and p-APNS adsorbed on the surface of aluminium according to the Langmuir adsorption isotherm, but Temkin also fits for p-APNS. Phenomenon of mixed adsorption is proposed from the values of free energy of adsorption obtained. Thermodynamic parameters reveal that the adsorption process is spontaneous. p-APNS was found to be a better inhibitor than o-APNS.

Keywords : Al-Pure, Schiff base, Weight loss method, Corrosion, Acid.

INTRODUCTION

The most central feature of aluminium is its corrosion resistance due to the presence of a thin, adherent and protective surface oxide film. Because of this advantage, aluminium and its alloys are widely used in many industries such as pipes, chemical batteries, machinery and reaction vessels [1]. Acid solutions are commonly used in the chemical industry to purge the scales from the metallic surfaces [2]. Hydrochloric acid and sulphuric acid solutions are used for pickling of aluminium or for its chemical or electrochemical etching [3]. The consumption of inhibitors is one of the most practical approach to protect metals against acid attack [4]. Various organic inhibitors have been premeditated as corrosion inhibitors [5]. The applicability of organic compounds as corrosion inhibitors for metals in acidic media has been recognized for a long time.

Inhibition of metal corrosion by organic compounds is a result of adsorption of organic molecules or ions at the metal surface materializing a protective layer. This layer reduces or prevents corrosion of the metal. The action of such inhibitors depends on the specific interaction between the functional groups and the metal surface. The extent of adsorption depends on the nature of the metal, the metal surface condition, the mode of adsorption, the chemical structure of the inhibitor and the type of corrosion media [6, 7]. Thus, many studies concerning the inhibition of Al corrosion using organic substances are conducted in acidic and basic solutions [7-12]. Recently we have published research articles based on the use of various Schiff bases as corrosion inhibitors for zinc in sulphuric acid [13-20] and hydrochloric acid [21] has been reported.

In the continuation of our research work on developing unique corrosion inhibitors, herein we report the corrosion of Al-Pure in hydrochloric acid, containing ortho-Aminophenol-N-Salicylidene and para-Aminophenol-N-Salicylidene. Due to the presence of an imine $>C = N-$ group and conjugated double bonds, these should function as good inhibitors. The effect of inhibitor concentration and temperature on the behavior of the Schiff bases has been investigated.

MATERIAL AND METHODS

Preparation of Aluminium

The sample selected for the study was pure aluminium. The 6.0 X 3.0 X 0.05 cm dimension of specimens was used for weight loss techniques. Prior to each experiment, the surface pre-treatment of Al specimens was performed by mechanical polishing (using a polishing machine) with jeweler rouge, which gave a mirror like finish. The specimen was further degreased by immersion in A.R. CCl4 (sulphur free) and finally stored in desiccators prior to use for weight loss measurements.

The corrosive solution (1.0 M HCl) was prepared by dilution of analytical grade 37% HCl (NICE) with double distilled water. The concentration range of employed inhibitor was 0.01 % to 0.8 % in 1.0 M HCl. The used chemicals for preparation of Schiff base were AR grade (MERCK).

Preparation of Schiff Base

In a 500 c.c. three necked round bottom flask provided with a mechanical stirrer, one mole of salicyladehyde is placed in a 200 c.c. 95% alcohol. Cool the reactants at 0-5°C by immersing the flask in ice-bath. Slowly one mole of o-hydroxy aniline is added with rapid stirring. The reaction mixture is refluxed in water-bath for one hour. The reaction mixture is kept for 15 minutes after completion of the reaction, it is then poured in ice-cold

distilled water. Crude solid product separated then washed with water. Crude product is further purified by recrystalization from 95% ethanol. The pure product is filtered on Buchner funnel, by suction and air-dried. The melting point of pure o-Aminophenol-N-Salicylidene is 185°C. Synonym of o-Aminophenol-N-Salicylidene (o-APNS) is Phenol-2-[(2-hydroxyphenyl)iminomethyl] and Phenol-o-[N-(o-aminophenyl)formimidoyl], which is orange crystalline substance, is insoluble in water but soluble in ethanol.

The chemical structure of used Schiff bases is shown below:
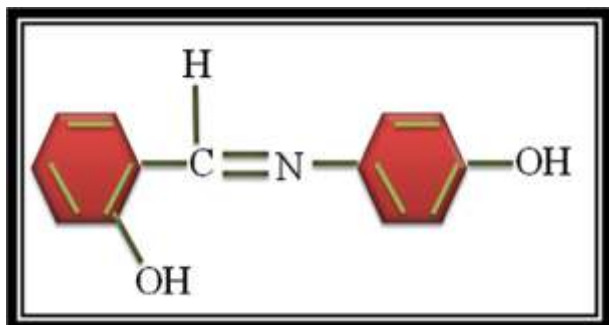
o-Aminophenol-N-Salicylidene
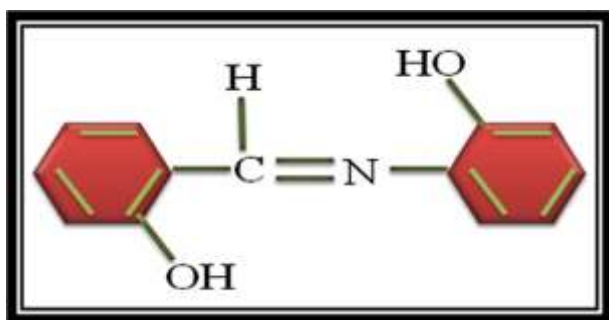
p-Aminophenol-N-Salicylidene

EXPERIMENTAL

Weight loss method was used to determine the corrosion inhibition efficiencies of o-APNS and p-APNS.

Weight Loss Method:

In weight loss method, the specimens were exposed to 1.0 M HCl solution containing controlled addition of inhibitor in the range of 0.01% to 0.8% inhibitor concentration. One specimen was suspended by a glass hook in each beaker containing 230 ml of the test solution prepared in 1.0 M HCl which was open to the air at 35° ± 0.5°C, to the same depth of about 1.5cm below the surface of the test solution. The experiments were repeated at different temperatures, ranging from 35° ± 0.5°C to 65° ± 0.5°C in absence and presence of 0.5% inhibitor's concentration after 30 minutes.

RESULTS AND DISCUSSION

Influence of Inhibitor Concentration on The Corrosion Rate and Inhibition Efficiency By Weight Loss Method

The corrosion rate of Al-Pure in absence and presence of inhibitors at 35° ± 0.5°C were studied using weight loss method. Table-1 display that the calculated values of inhibition efficiency (%IE) and surface coverage (θ) for Al-Pure dissolution in 1.0 M HCl in absence and presence of inhibitors. The results show that inhibitors actually inhibited the corrosion of Al-Pure in 1.0 M HCl solutions. The inhibition efficiency was found to be depended on the concentration of the inhibitors. As the concentration of the inhibitors increases, the inhibition efficiency (%IE) is going up to a maximum values. The inhibitory action of the inhibitors against Al-Pure corrosion can be attributed to the adsorption of Schiff base molecules on the Al-Pure surface, which limits the dissolution of the latter by blocking it corrosion sites and hence decreasing the weight loss, with increasing efficiency as the concentration increases. There have been few research articles in literature described similar kind of investigation [22, 23].

Table-1 Corrosion parameters for Al-pure in the presence and absence of different concentrations of o-APNS and p-APNS obtained from weight loss measurement at 350C ± 0.50C for exposure period of 30 minutes.

Inhibitor	Concentration (%W/V)	Weight loss (mg/dm²)	Surfac coverage (θ)	(%IE)
Blank	-	379	-	-
o-APNS	0.01	84.0	0.779	77.9
	0.05	24.4	0.936	93.6
	0.10	19.0	0.950	95.0
	0.15	8.1	0.979	97.9
	0.20	8.1	0.979	97.9
	0.50	5.4	0.986	98.6
	0.80	2.7	0.993	99.3
p-APNS	0.01	21.7	0.943	94.3
	0.05	16.3	0.957	95.7
	0.10	10.8	0.971	97.1
	0.15	5.4	0.986	98.6
	0.20	5.4	0.986	98.6
	0.50	2.7	0.993	99.3
	0.80	2.7	0.993	99.3

The inhibition efficiency (%IE) and degree of surface coverage (θ) were calculated using equations 1 and 2, respectively [24]:

$$\text{Inhibition efficiency (\%IE)} = \frac{Wu - Wi}{Wu} \times 100 \quad ....(1)$$

$$\text{Surface coverage}(\theta) = \frac{Wu - Wi}{Wu} \quad ....(2)$$

Where 'Wu' and 'Wi' are the weight loss of Al-Pure in 1.0 M HCl in absence and presence of inhibitor.

Adsorption Behavior

The transition of metal/solution interface from a state of active dissolution to the passive state can be attributed to the adsorption of the inhibitor molecules at the metal/solution interface, forming a protective film. The rate of adsorption is usually rapid and hence, the

reactive metal surface is shielded from the aggressive environment. It is well recognized that organic inhibitor molecules set up their inhibition action via the adsorption of the inhibitor molecules onto the metal/solution interface. Adsorption process can occur through the replacement of solvent molecules from metal surface by ions and molecules accumulated in the vicinity of metal/solution interface. Aromatic compounds (which contain the benzene ring) undergo particularly strong adsorption on many electrode surfaces. The bonding can occur between metal surface atoms and the aromatic ring of the adsorbate molecules or ligands substituent groups. The exact nature of the interactions between a metal surface and an aromatic molecule depends on the relative coordinating strength towards the given metal of the particular groups present.

The values of surface coverage (θ) (given in Table-1) for the different concentrations of the studied inhibitor at 35 ± 0.5 °C have been used to explain the best adsorption isotherm to determine the adsorption process. Attempts were made to fit θ values of various isotherms including Langmuir, Temkin and Freundlich isotherms. Assessment of the values of o-APNS reveals that the inhibitor (o-APNS) obeys Langmuir adsorption isotherm [25] and assessment of the values for p-APNS reveals that the experimental data fitted with the Langmuir adsorption isotherm, means that there is no interaction between the adsorbed species. This inhibitor also obeys Temkin adsorption isotherm which fits superior than the Langmuir adsorption isotherm [26].

Langmuir adsorption is shown by the equation:

$$\log C_{inh} = \log \frac{1}{K} + \log \frac{\theta}{1-\theta} \qquad .....(3)$$

Temkin adsorption is shown by the equation:

$$\theta = \log \frac{1}{a} C_{inh} + cons \tan t \qquad .....(4)$$

Where " is the degree of surface coverage, 'C_{inh}' is the concentration of the inhibitor; 'K' is the equilibrium constant for adsorption process.
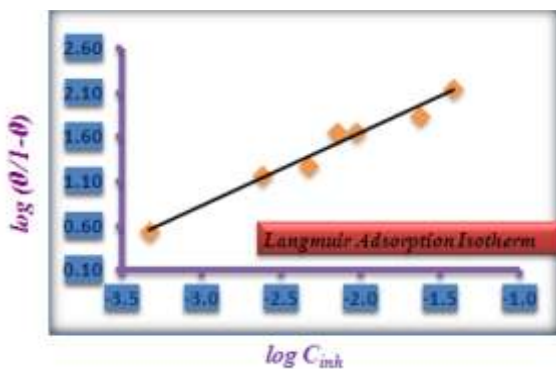

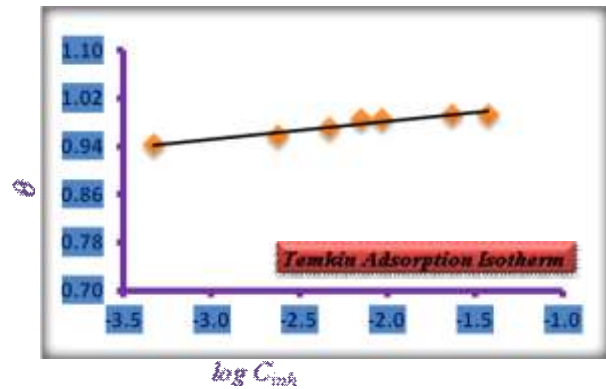**Figure-1: Langmuir adsorption isotherm plot for o-APNS at 35° C for 30 minutes.**

Figure-2: Langmuir and Temkin adsorption isotherm plot for p-APNS at 35° C for 30 minutes

Effect of Temperature

The effect of temperature on inhibition efficiency was studied at various temperatures ranging from 35oC – 65oC for 30 minutes in presence and absence of inhibitors by weight loss technique. The effect of temperature change for exposure period of 30 minutes on corrosion inhibition of Aluminium by inhibitors is shown in Figure-3 and Figure-4 for o-APNS and p-APNS respectively. The results from Table-2 show that inhibition efficiency decreases with increase in temperature. These results suggest that there is an increase in rate of metal dissolution and hence the equilibrium is shifted to inhibitor desorption leading to decrease in surface coverage degree.

Table-2. Effect of temperature on weight loss and inhibition efficiency for Al-Pure in 1.0 M HCl for 30 minutes.

Inhibitor concentration (%w/v)	Weight loss (mg/dm^2) at a temperature of			
	35^oC	45^oC	55^oC	65^oC
Nil(HCl only)	379	2439	3794	4743
o-Aminophenol-N-Salicylidene (o-APNS)				
0.5	5 (98.6%)	54 (97.8%)	407 (89.3%)	1084 (77.1%)
p-Aminophenol-N-Salicylidene (p-APNS)				
0.5	3 (99.3%)	22 (99.1%)	79 (97.9%)	279 (94.1%)

Values in brackets represents the percent inhibition.

Thermodynamic Parameters

Activation energy Ea, heat of adsorption Qads, Gibb's free energy of adsorption Gads, these parameters attributes to the type of adsorption occurring in the corrosion inhibition process.

In acidic solution the corrosion rate is related to temperature by Arrhenius Equation 5.

$$\rho = k \exp^{(\div EaRT} \qquad ..... (5)$$

Where 'ρ' is the corrosion rate, 'Ea' is the apparent activation energy, 'R' is molar gas constant and 'T' is the

absolute temperature. The apparent activation energy for exposure period of 30 minutes is determined from the slope of log ρ versus $1/T \times 10^4$ graph depicted in Figure-3 and Figure-4 for o-APNS and p-APNS inhibitors.
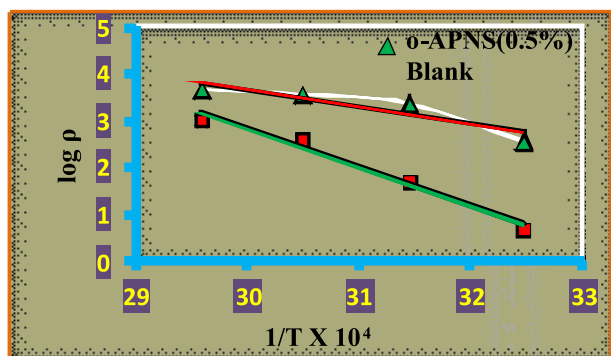

Figure-3 Plotting log ρ vs. $1/T \times 10^4$ to calculate the apparent Activation Energy of corrosion process in the presence and absence of o-APNS.
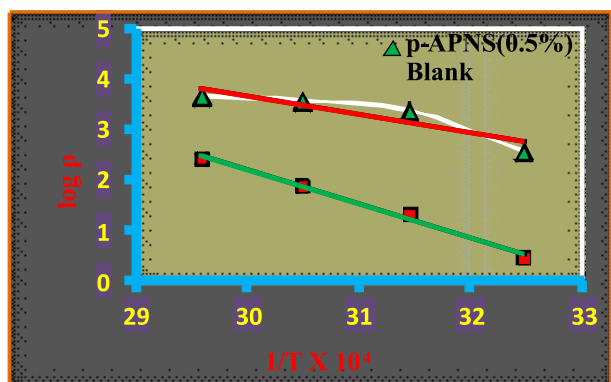

Figure-4 Plotting log ρ vs. $1/T \times 10^4$ to calculate the apparent Activation Energy of corrosion process in the presence and absence of p-APNS.

The relationship between the temperature, inhibition efficiency (I.E %) of an inhibitor and the apparent activation energy in the presence of on inhibitor was given as follows:

The value of apparent activation energy 'E_a' of inhibited solution was found to be greater than that of uninhibited solution for those inhibitor whose I.E% decreases with the increase in temperature. This assumption is confirmed by calculating the apparent activation energy from the slope of Figure-3 and Figure-4 for o-APNS and p-APNS, respectively. The calculated apparent activation energy is shown in Table-3 [28]. If it is assumed that the inhibitor is adsorbed on the metal surface in the form of a monolayer film, covering at any instant a fraction, , of the metal surface in a uniform random manner, then the heat of adsorption, Q_{ads}, of the inhibitor can be calculated from the equation:

$$Q_{ads} = 2.303 \, R \left(log \frac{\theta_2}{1-\theta_2} - log \frac{\theta_1}{1-\theta_1} \right) \left(\frac{T_1 T_2}{T_2 - T_1} \right) \quad ....(6)$$

Where 'θ_2' and 'θ_1' are the values of surface coverage at temperatures T_2 and T_1 respectively.

The values of free energy of adsorption were calculated from Equation 7 and 8

$$logC_{inh} = log\frac{\theta}{1-\theta} - log B \qquad .....(7)$$

$$where, \; logB = 1.74 - \frac{\Delta G_{ads}}{2.303 \, RT} \qquad .....(8)$$

The values of ΔG_{ads} and Q_{ads} are shown in Table-3. The negative value of ΔG_{ads} indicates that the inhibitor molecules are strongly adsorbed on the metal surface, the values also indicate a spontaneous adsorption of the inhibitor molecules usually characterized by the strong interactions with the metal surface. Generally, the values of ΔG_{ads} around -20 kJ/mol or lower are consistent with physisorption, while those around -40 kJ/mol and above involve chemisorptions. In the present work, the calculated value of ΔG_{ads} are almost slightly less negative than -40 kJ/mol indicating that the adsorption of inhibitor molecule is not merely physisorption or chemisorptions but obeying a comprehensive adsorption (physical and chemical adsorption). It was observed, limited decrease in the absolute value of (ΔG_{ads}) with an increase in temperatures, indicating that the adsorption was somewhat unfavorable with increasing experimental temperature, indicating that physisorption has the major contribution while chemisorption has the minor contribution in the adsorption mechanism. The negative value of the heat of adsorption (Q_{ads}) indicates that the adsorption is exothermic in nature. This observation further confirms physical adsorption of the inhibitor on the metal surface in HCl solution.

Table-3: Apparent Activation energy (Ea), heat of adsorption (Qads) and free energy of adsorption (Gads) for adsorption of o-APNS and p-APNS inhibitor on pure Aluminium in 1M HCl for exposure period of 30 minutes

Inhibitor	*E_a* *KJ/mol*	*ΔG_{ads}* *KJ/mol*	*Q_{ads}* *KJ/mol*
Blank	70.3	-	-
o-APNS	157.7	-28.3	-94.2
p-APNS	129.1	-31.6	-61.7

Mechanism of Adsorption and Inhibition

The transition of metal/solution interface from a state of active dissolution to the passive state can be attributed to the adsorption of the inhibitor molecules at the metal/solution interface, forming a protective film. Adsorption process can occur through the replacement of solvent molecules from metal surface by ions and molecules accumulated in the vicinity of metal/solution interface. Aromatic compounds (which contain the benzene ring) undergo particularly strong adsorption on many electrode surfaces. The bonding can occur between metal surface atoms and the aromatic ring of the adsorbate molecules or ligands substituent groups. The exact nature

of the interactions between a metal surface and an aromatic molecule depends on the relative coordinating strength towards the given metal of the particular groups present.

The adsorption of Schiff base compound (SB) at Al-Pure/hydrochloric acid interface can be attributed to the presence of hetero atoms (N, O and S), azomethine group and aromatic rings. Thus, the possible reaction centres are unshared electron pair of hetero-atoms and -electrons of azomethine group and aromatic rings. The adsorption of SB at Al-Pure /1.0 M HCl interface can be explained as follows. Schiff base molecules might be protonated in the acid solution as

$$C_{13}H_{11}NO_2 + xH^+ \rightarrow [C_{13}H_{11}NO_2H_x]^{x+} \quad .....(9)$$
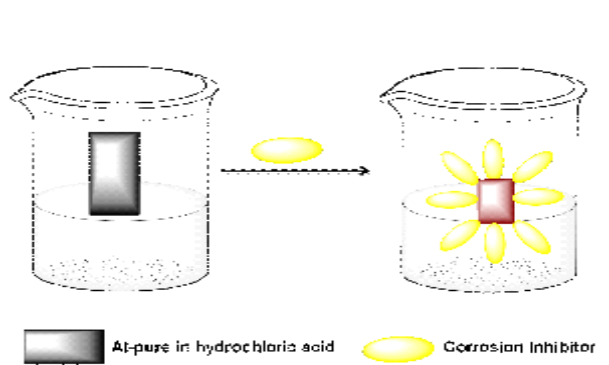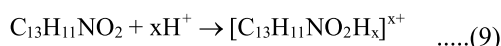
In aqueous acidic solutions, the SB molecules exist either as neutral molecules or in the form of protonated molecules (cations). SB may adsorb on the metal/acid solution interface by one and/or more of the following ways: (i) electrostatic interaction of protonated Schiff base compound with already adsorbed chloride ions (synergistic effect), (ii) donor–acceptor interactions between the -electrons of aromatic ring and vacant p orbital of surface aluminium atoms, (iii) interaction between unshared electron pairs of hetero atoms and vacant p-orbital of aluminium surface atoms [28].

CONCLUSION:

Our present study leads to the following conclusions for controlling the corrosion of pure Aluminium by Schiff base in 1 M HCl:

1. SB acted as an efficient inhibitor for the corrosion of Al-Pure in 1.0 M HCl and exhibited 99.3% efficiency at concentration of 0.8% w/v for o-APNS and at 0.5% w/v for p-APNS.

2. The adsorption follows Langmuir isotherm for o-APNS and for p-APNS the adsorption follows both Langmuir and Temkin isotherm, but temkin fits better than Langmuir isotherm.

3. As temperature increases, the inhibition efficiency decreases for both the inhibitors.

4. The heat of adsorption and free energy of adsorption are negative while the entropy of adsorption is positive. The free energy of adsorption are more negative in case of p-APNS inhibitor and the values shows that the inhibitors are of mixed type. The negative values of heat of adsorption suggest that the adsorption is an exothermic and the positive values of entropy of adsorption suggest that the adsorption is a spontaneous process.

5. Apparent activation energy is higher in the presence of inhibitor compared to that in plain acid.

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