

# **Electrochemical and Adsorption Behaviour Study for Corrosion Inhibition Efficiency and Position of Group in Schiff Base for Aluminium in Acidic Media**

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**Abstract:** Inhibition efficiency of newly synthesized Schiff base of benzaldehyde were investigated based on the position of functional group towards Aluminium in HCl using weight loss and electrochemical techniques –polarization and electrochemical impedance spectroscopy (EIS). Inhibition efficiency of these inhibitors was found to depend on the adsorption behaviour and molecular structure remarkably when the inhibitor concentration is low. Para substituted molecule has shown high inhibition efficiency in general.

**Keywords:** o, p- substituted Schiff base, Corrosion inhibition, EIS.

## **I. INTRODUCTION**

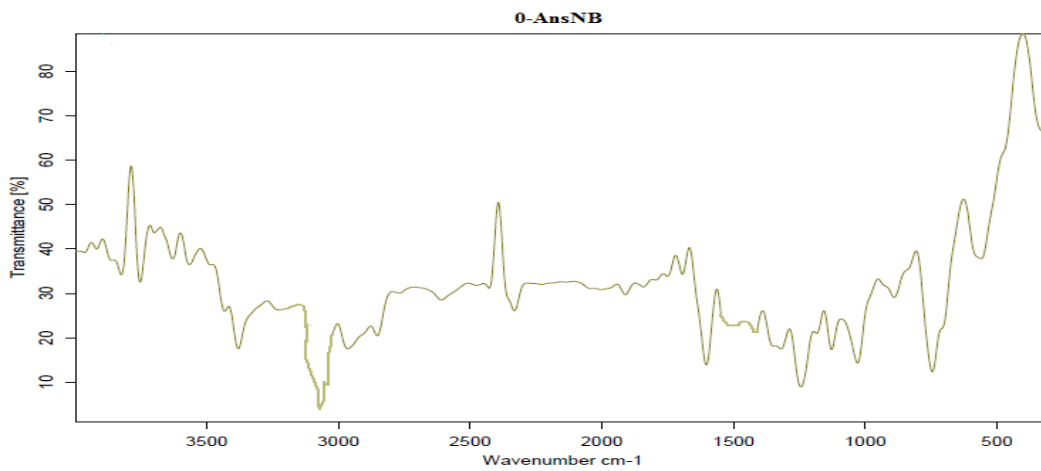
Corrosion is the deterioration of metal by chemical attack or reaction with its environment. Corrosion processes develop fast after disruption of the protective barrier and are accompanied by a number of reactions that change the composition and properties of both the metal surface and the local environment. The study of corrosion of metal and its alloys is a matter of tremendous theoretical and practical concern and as such has received a considerable amount of interest [1]. Aluminium and its alloys are very good corrosion resistant materials in neutral aqueous solution, due to the formation of passive film and are remarkable economic and attractive materials for engineering applications owing to its low cost, light weight, high thermal and electrical conductivity [2]. It is well known that pitting corrosion occurs on metals covered with passive films. The corrosion of metal is the result of two simultaneous reactions that are in electrical equilibrium, (i) Oxidation of metal to metal ions, (ii) Reduction of hydrogen to hydrogen gas. Hydrochloric acid and sulphuric acid solutions are used for pickling of aluminium for its chemical or electrochemical etching. It is very important to add corrosion inhibitors to decrease the rate of metal dissolution in such solutions [3]. Inhibition of metal corrosion by organic compounds is a result of adsorption of organic molecules or ions at the metal surface forming 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 [2, 4]. Thus, many studies concerning the inhibition of Al corrosion using organic substances are conducted in acidic and basic solutions [3-9].

In the present work the corrosion of Al-Pure in hydrochloric acid, containing ortho- & para-Anisidine-N-benzylidene has been reported. 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, exposure period and temperature on the behavior of the Schiff base has been investigated.

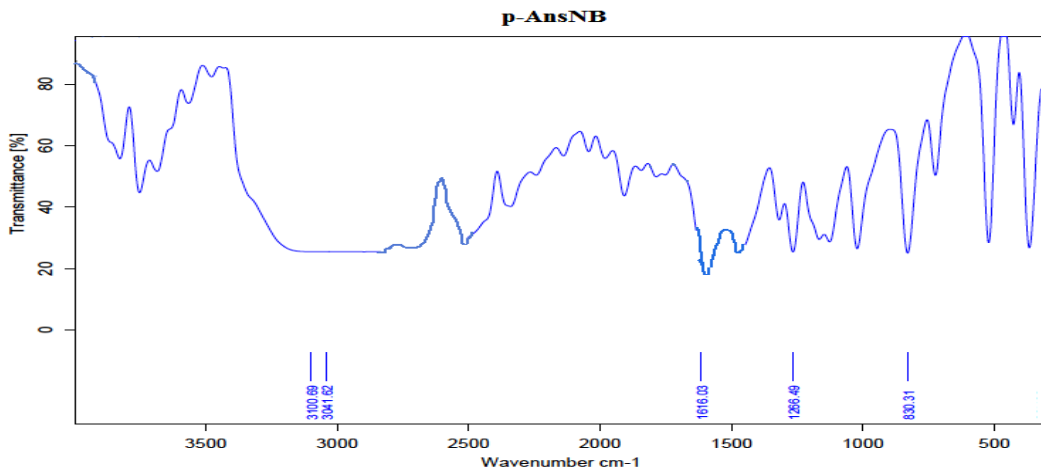
**II. EXPERIMENTAL**

*2.1 Synthesis of Schiff base*

The Schiff's base was synthesized from equimolar amount of benzaldehyde with respective amines o-Anisidine (o-Ans) and p-Anisidine (p-Ans) through a condensation reaction in the methanol media as per described by Shah et al [10]. The chemical structure of used Schiff's base is characterized by IR (fig.1), which is shown below:



[A]  
aromatic ring:- 3090  $\text{cm}^{-1}$ ,  $>\text{C}=\text{N}$  :- 1623  $\text{cm}^{-1}$ ,  $-\text{C}-\text{OCH}_3$  :- 1279  $\text{cm}^{-1}$ ,  
o-disubstituted:- 747  $\text{cm}^{-1}$



[B]  
aromatic ring:- 3040 -3100  $\text{cm}^{-1}$ ,  $>\text{C}=\text{N}$  :- 1616  $\text{cm}^{-1}$ ,  $-\text{C}-\text{OCH}_3$  :- 1266  $\text{cm}^{-1}$ ,  
p-disubstituted:- 830  $\text{cm}^{-1}$

Fig. 1 IR spectra [A]- o-AnsNB and [B]-p-AnsNB

*2.2 Electrodes and electrolytes:*

Rectangular specimens of Al-Pure, of size 6.0cm  $\times$  3.0 cm (thickness = 0.050 cm), with a small hole of  $\sim$  2mm diameter just near one end of the specimens were used for the determination of corrosion rate in weight loss method. The specimen

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was polished to mirror finish using successively grade Oakey emery followed by jeweler's rough. The specimens were finally degreased with A.R. carbon tetrachloride (sulphur free).

For polarization and impedance measurements, metal coupons of circular design, diameter 2.802 cm with a handle length of 3 cm × 0.5 cm with the thickness 0.087 cm with small hole of about 2 mm diameter just near the upper end of the specimen were used. The handle and the back of the coupon and of the auxiliary platinum electrode were coated with Perspex leaving only the circular portion of the specimen of apparent surface area 6.156 cm<sup>2</sup> exposed to the solution.

The corrosive solutions (1.0 M) were prepared by dilution of analytical grade 37% HCl (NICE) with double distilled water. The concentration range of employed inhibitor was 0.0001% to 0.8 % in 1.0 M HCl. The used all chemicals for preparation of Schiff's base were AR grade (MERCK).

## 2.3 Measurements:

Three methods namely weight loss method, impedance spectroscopy and polarization study was used to determine the corrosion inhibition efficiencies of o-AnsNB and p-AnNB.

### 2.3.1 Weight loss method:

In weight loss method, the test specimens were immersed in 230 ml of 1.0 M hydrochloric acid solution without and with various concentrations of inhibitors in the range 0.0001% to 0.8%. One specimen only, was suspended by a Pyrex glass hook in each beaker containing 230 ml of the test solution which was open to the air at 35° ± 0.5° C (unless otherwise specified ) for different exposure period, to the same depth of about 1.5 cm below the surface of the solution. The weight of the specimen before and after immersion was determined using mettler balance – M5 type. The accuracy of the balance was 0.0001gm. Triplicate experiments were performed. After completing weight loss experiments the metal coupon was washed with double distilled water, dried and weighed. Experiment (the loss in weight of specimen ) were carried out at 0.05%, 0.5% & 0.8% concentration of inhibitor at temperature ranging from 35° ± 0.5° C to 65° ± 0.5° C.

### 2.3.2 Electrochemical measurements

Electrochemical experiments were carried out using a standard electrochemical three electrode cell assembly, Al-Pure was used as working electrode, platinum as counter electrode and saturated calomel electrode (SCE) as reference electrode. The test solution was contained in H-type (80 ml in each limb) Pyrex glass cell with Luggin capillary as near to the electrode surface as possible and porous partition to separate the two compartments.

(a) The potential was measured against a saturated calomel electrode (SCE), in polarization study. The corrosion parameters such as corrosion potential ( $E_{corr}$ ), corrosion current density ( $I_{corr}$ ) and Tafel plots were measured by polarization method. In this study, the current density was varied in the range of  $2 \times 10^{-4}$  to  $3.25 \times 10^{-4}$  A cm<sup>-2</sup>.

(b) Electrochemical impedance measurements were carried out in the frequency range of 20 kHz – 0.1 Hz at the open circuit potential (OCP), after 30 minutes of immersion by applying amplitude of 5 mV sine wave ac signal (AUTOLAB). Double layer capacitance ( $C_{dl}$ ) and charge transfer resistance ( $R_{ct}$ ) values were calculated from Nyquist plots as described by Hosseine [7]. EIS data were analyzed using frequency response analyzer (FRA) electrochemical setup.

## III. RESULTS AND DISCUSSION

### 3.1 Weight Loss

Corrosion of any metal results in the loss in weight of the metal; hence weight loss measurement is the standard method. Weight loss measurement for both the inhibitors performed in absence and presence of different inhibitor concentration.

#### a) Effect of inhibitor concentration and adsorption isotherm

Weight loss results obtained for the Aluminium immersed in 1M HCl solution containing different concentrations of each inhibitor. The corrosion rate, surface coverage and inhibition efficiency calculated from it are shown in (Table –I). The results show that at the lower inhibitor concentration there is considerable difference in inhibition efficiency for o-AnsNB and p-AnsNB and inhibition efficiency of p-AnsNB is remarkably higher than o-AnsNB, hence we can say that the inhibition efficiency of these compounds depends on the position of –OCH<sub>3</sub> group. But both the inhibitor reduced the corrosion rate to a significant extent when inhibitor concentration is (0.001% w/v) or more and at the optimum inhibitor

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concentration they offered equal efficiency (99.78%) proves that both the inhibitors are excellent inhibitors. It can be seen that inhibition efficiency increases with increase in inhibitor concentration which suggest that inhibition is a result of adsorption of inhibitor on the metal surface. [11].

TABLE 1  
WEIGHT LOSS DATA FOR ALUMINIUM IN 1M HCl AT 35 ± 0.5° C

Inhibitor	Inhibitor concentration (w/v %)	Weight loss (mg/dm <sup>2</sup> )	Surface Coverage (θ)	Inhibition efficiency (%)
HCl only	-	2312	-	--
<i>o-AnsNB</i>	1 × 10 <sup>-4</sup>	2138	0.0727	7.27
	5 × 10 <sup>-4</sup>	1358	0.4118	41.18
	1 × 10 <sup>-3</sup>	561	0.7574	75.74
	5 × 10 <sup>-3</sup>	249	0.8923	89.23
	5 × 10 <sup>-2</sup>	89	0.9615	96.15
	5 × 10 <sup>-1</sup>	8	0.9965	99.65
	8 × 10 <sup>-1</sup>	5	0.9978	99.78
<i>p-AnsNB</i>	1 × 10 <sup>-4</sup>	1314	0.4317	43.17
	5 × 10 <sup>-4</sup>	1018	0.5597	55.97
	1 × 10 <sup>-3</sup>	224	0.9031	90.31
	5 × 10 <sup>-3</sup>	177	0.9234	92.34
	5 × 10 <sup>-2</sup>	51	0.9779	97.79
	5 × 10 <sup>-1</sup>	5	0.9978	99.78
	8 × 10 <sup>-1</sup>	5	0.9978	99.78

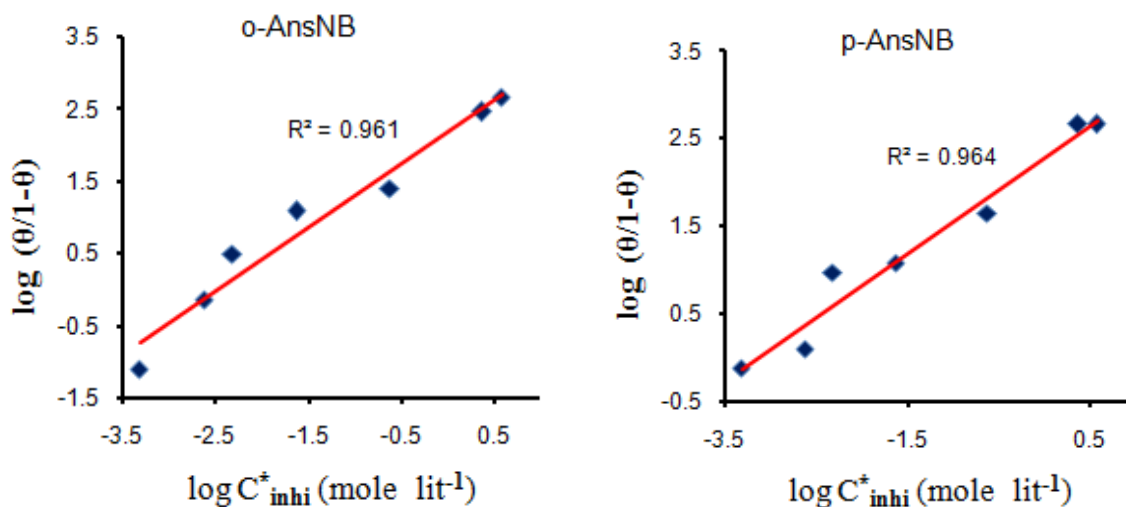


Fig. 2 Langmuir Adsorption isotherm at 35 ± 0.5° C for Aluminium in 1M HCl solution

Adsorption isotherms are often shown to demonstrate the performance of organic adsorbent type inhibitors and important in determining the mechanism of organic electrochemical reactions. The adsorption of inhibitor at the metal/solution interface is the first step in the inhibition mechanism. There are three types of possibilities for adsorption that may take

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place in case of organic molecule: (i) electrostatic attraction between the charged metal and the charged molecules (Physisorption) (ii) interaction of  $\pi$ - electrons with the metal (Chemisorption), (iii) interaction of uncharged electrons' pair in the molecule with the metal, and (iii) combination of (i), (ii) and (iii) [12]. The nature and description of the isotherm was explained by using the values of surface coverage ( $\theta$ ). Amongst the most frequently used isotherms, the Langmuir adsorption isotherm provides the best description of adsorption behaviour (Figure-2)

### b) Effect of temperature on the performance of Schiff base inhibitor

To study the effect of temperature on the inhibition efficiency, weight loss measurements of Aluminium specimens were carried out in absence and presence of 0.05%, 0.5% & 0.8% (w/v) concentration of inhibitors at temperature ranging from  $35^\circ\text{C} \pm 0.5^\circ\text{C}$  to  $65^\circ\text{C} \pm 0.5^\circ\text{C}$ . Corrosion rate and (IE %) was calculated. Inhibition efficiencies were found to decrease with increase in temperature. The decrease in inhibition efficiency with increasing temperature may be due to desorption of some adsorbed molecule from the metal surface at higher temperatures. But at optimum concentration of inhibitor, inhibition efficiency of both the inhibitor is good which can be associated with the combination of physisorption and chemisorption for these molecules and it is observed that p-AnsNB offer higher inhibition than o-AnsNB even at higher temperature may be due to the presence of  $-\text{OCH}_3$  group in the Para position. Thermodynamic parameters are listed in (Table-2).

TABLE 2  
THERMODYNAMIC PARAMETERS FOR THE INVESTIGATED SCHIFF BASES

Inhibitor	Inhibitor Concentration (% w/v)	E <sub>ads</sub> (kJ mol <sup>-1</sup> )	ΔG <sup>0</sup> <sub>ads</sub> (kJ mol <sup>-1</sup> )	Q <sub>ads</sub> (kJ mol <sup>-1</sup> )
<b>Hcl Only</b>				
<b>o-AnsNB</b>	0.05	115.261	-28.94	-161.694
	0.5	112.002	-32.37	-90.668
	0.8	61.268	-34.91	-44.431
<b>p-AnsNB</b>	0.05	132.3	-31.2	-171.62
	0.5	49.97	-33.4	-23.85
	0.8	43.27	-36.07	-17.84

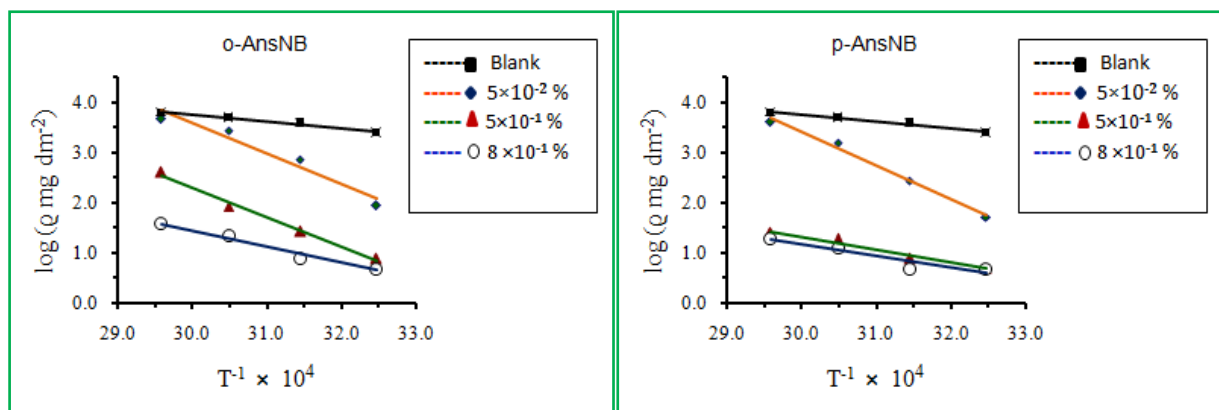


Fig. 3 Arrhenius plots for Aluminium in 1M HCl [A] o-AnsNB & [B] p-AnsNB

Thermodynamically, the negative  $\Delta G_{ads}^0$  value indicates the adsorption process is a spontaneous process, and inhibitor form a stable adsorbed layer on the metal surface. In general it is known that physical adsorption, lead to less negative

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$\Delta G_{ads}^0$  value than -20 kJ/mol. On the other hand chemical adsorption leads to more negative  $\Delta G_{ads}^0$  value than -40 kJ/mol [13]. Thus, the  $\Delta G_{ads}^0$  values obtained in the present work suggest that the adsorption of both the inhibitor may be initiated as physical adsorption and chemical adsorption accompanied the adsorption process leading to the good inhibition efficiency at higher temperature with (0.8% w/v) inhibitor concentration. In addition, slightly more negative  $\Delta G_{ads}^0$  values of p-AnsNB the interaction between p-AnsNB and metal surface is slightly stronger than those between o-AnsNB and a metal surface. The negative value of  $Q_{ads}$  indicates that the adsorption of used inhibitor on the aluminium surface is exothermic.

### 3.2 Electrochemical Impedance Spectroscopy

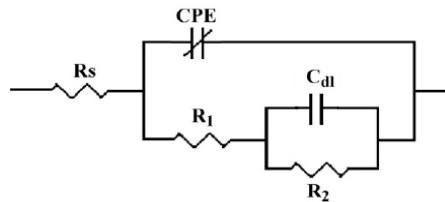


Fig 4. The Equivalent Circuit Model used to fit the Experimental Results

The impedance spectra were analysed by fitting to the equivalent circuit model as shown in (figure 4). For the Al/acid interface model, this circuit is generally used to describe EIS data. In this circuit  $R_s$  is solution resistance,  $R_{ct}$  charge transfer resistance and CPE is constant phase element. The CPE parameters were used to derive double layer capacitance ( $C_{dl}$ ) values by use of the following equation.

$$C_{dl} = \frac{1}{\omega \cdot R_{ct}} \quad \text{Here } \omega \text{ is angular frequency.}$$

$$\omega = 2\pi f_{max}$$

Where,  $f_{max}$  is the frequency at which the imaginary component ( $Z_{imag}$ ) of the impedance has maximum value. The inhibitor efficiency of the inhibitors from impedance data was computed by using the following equation:

$$(\% \text{ IE}) = \frac{R_{ct} - R_{ct}^0}{R_{ct}} \times 100$$

Where,  $R_{ct}^0$  and  $R_{ct}$  are charge transfer resistance for aluminium in 0.1M HCl in the absence and presence of inhibitors respectively. The parameters deduced from the impedance technique are given in Table 3.

The impedance results obtained for pure Aluminium in 1M HCl, at frequency range of 20 KHz –0.1 Hz, in the presence and absence of inhibitors are presented as Nyquist plots in (figure 5[A] and 5[B]) respectively. As shown in the Figure 5, Nyquist plots obtained with increase in inhibitor concentration are in the form of a depressed semicircle with increase in diameter.

A single semicircle has been obtained over the frequency range studied, shifted along the real impedance axis ( $Z_{real}$ ), which can be attributed to charge transfer of the corrosion process. This indicates that with increasing amount of inhibitor, the inhibitor formed more densely packed monolayer.

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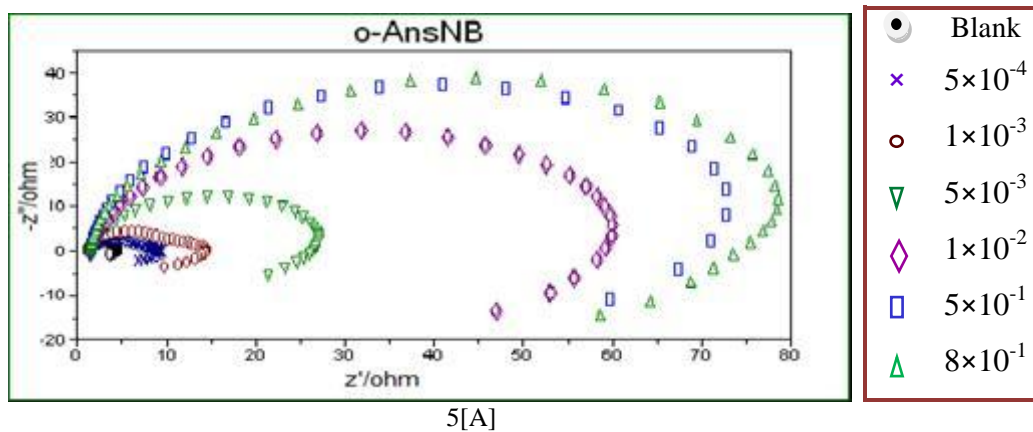
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TABLE 3  
IMPEDANCE PARAMETERS

Inhibitor	Inhibitor Concentration (%)	R <sub>s</sub> (ohm)	R <sub>ct</sub> (ohm)	C <sub>dl</sub> (μF)	(% ) IE	
					EIS	Wt.loss
Blank	-	1.256	3.46	113.1	-	-
	5×10 <sup>-4</sup>	1.581	6.63	112.6	47.81	41.18
	1×10 <sup>-3</sup>	1.562	11.30	109.3	69.38	75.74
	5×10 <sup>-3</sup>	1.605	32.6	91.4	89.39	89.23
	1×10 <sup>-2</sup>	1.608	42.4	87.4	91.84	90.40
	5×10 <sup>-1</sup>	1.678	70.6	65.82	95.1	99.65
	8×10 <sup>-1</sup>	1.527	78.6	48.97	95.6	99.78
o-AnsNB	5×10 <sup>-4</sup>	1.409	8.12	112.2	57.39	55.97
	1×10 <sup>-3</sup>	1.440	32.4	104.1	89.32	90.31
	5×10 <sup>-3</sup>	1.274	56.0	95.21	93.82	92.34
	1×10 <sup>-2</sup>	1.247	64.8	92.76	94.66	95.59
	5×10 <sup>-1</sup>	1.432	73.1	75.99	95.27	99.78
	8×10 <sup>-1</sup>	1.430	78.8	51.54	95.61	99.78

The observed deviations from perfect semicircles are often referred to the frequency dispersion of interfacial impedance. This anomalous phenomenon is interpreted as reflecting inhomogeneity of electrode surfaces arising from surface roughness or interfacial origin such as those found in adsorption processes.



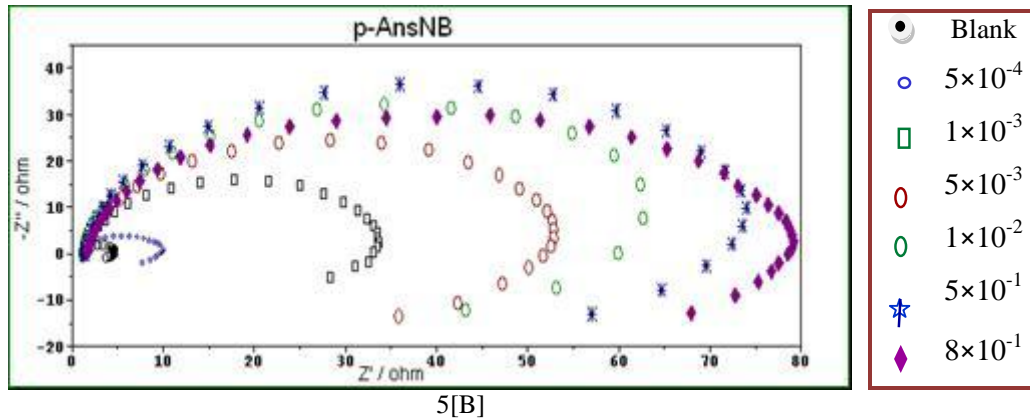


Fig. 5 Nyquist plots of Aluminium in 1M HCl solutions in the presence and the absence of inhibitors  
[A] o-AnsNB & [B] p-AnsNB

### 3.3 Polarization Studies

The polarization curves obtained for Aluminium in 1M HCl without and with different concentration of inhibitor are shown in the Figure 6 and Various parameters such as corrosion potential ( $E_{corr}$ ), corrosion current density ( $I_{corr}$ ), cathodic Tafel slop ( $b_c$ ), anodic Tafel slop ( $b_a$ ) and percentage inhibition efficiency (IE%) obtained from the measurement are given in Table 4.

TABLE 4

Electrochemical parameters of corrosion of Aluminium obtained from polarization method.

Inhibitor	Inhibitor Concentration (%)	$E_{corr}$ (mV)	$b_c$ (mV/dec)	$b_a$ (mV/dec)	$I_{corr}$ Cathodic ( $A\ cm^{-2}$ )	(%IE)
o-AnsNB	Blank	-814	0.123	0.137	$3.715 \times 10^{-3}$	-
	$5 \times 10^{-4}$	-818	167	098	$1.59 \times 10^{-3}$	57.34
	$1 \times 10^{-3}$	-818	0.147	0.114	$6.61 \times 10^{-4}$	82.22
	$5 \times 10^{-3}$	-819	0.151	0.103	$3.47 \times 10^{-4}$	90.67
	$1 \times 10^{-2}$	-828	0.145	0.106	$2.82 \times 10^{-4}$	92.41
	$5 \times 10^{-1}$	-836	0.171	0.091	$0.724 \times 10^{-4}$	98.05
	$8 \times 10^{-1}$	-842	0.177	0.098	$0.661 \times 10^{-4}$	98.22
p-AnsNB	$5 \times 10^{-4}$	-820	0.167	0.129	$1.585 \times 10^{-3}$	57.34
	$1 \times 10^{-3}$	-823	0.158	0.130	$7.59 \times 10^{-4}$	79.58
	$5 \times 10^{-3}$	-828	0.165	0.150	$7.08 \times 10^{-4}$	80.95
	$1 \times 10^{-2}$	-838	0.179	0.131	$6.46 \times 10^{-4}$	82.62
	$5 \times 10^{-1}$	-854	0.155	0.120	$3.236 \times 10^{-5}$	99.13
	$8 \times 10^{-1}$	-856	0.147	0.116	$2.951 \times 10^{-5}$	99.21



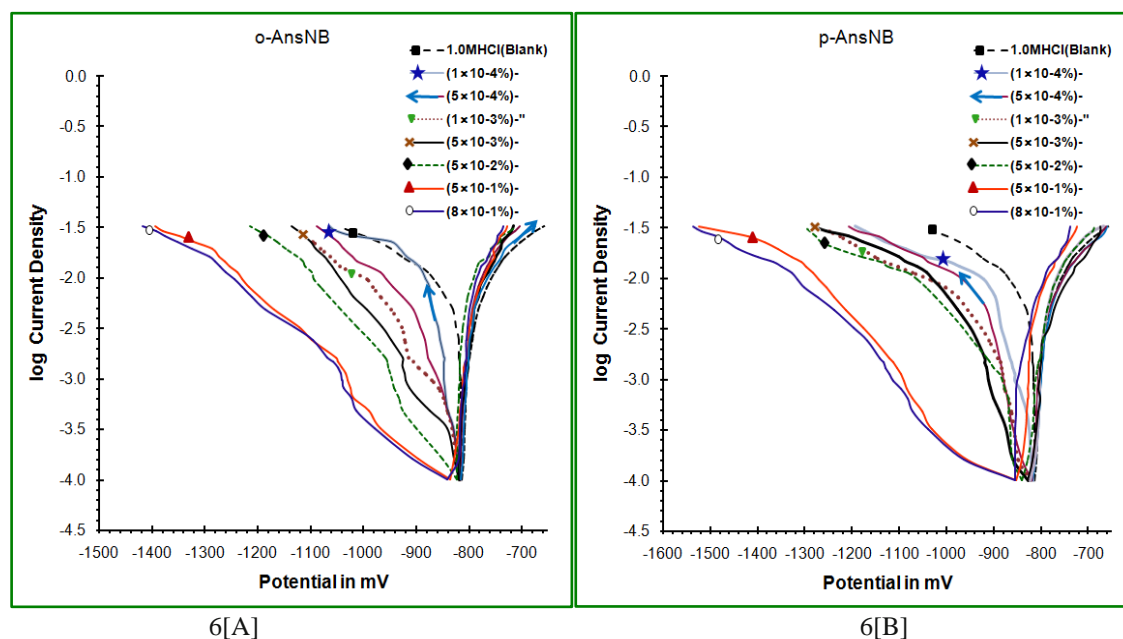


Fig. 6: Cathodic and Anodic polarization graphs for Aluminium in absence and presence of  
(A) *o*-AnsNB and (B) *p*-AnsNB respectively

The results show that in presence of inhibitor, value of  $E_{\text{corr}}$  shifted towards more negative direction and the value of  $I_{\text{corr}}$  decreases with increase in inhibitor concentration. The decrease in  $I_{\text{corr}}$  suggests decrease in electrochemical corrosion reaction and may be attributed to the formation of barrier layer over the aluminium surface by the inhibitor molecule. Though the value of  $E_{\text{corr}}$  shifted towards more negative but not significantly, which show that the addition of studied Schiff base affected both anodic and cathodic reaction, hence the Schiff base *o*-AnsNB and *p*-AnsNB are mixed type inhibitor.

#### IV CONCLUSION

1. The inhibition efficiency of Schiff base mainly attributed to  $>C=N$  group, and the presence of  $-OCH_3$  group in a molecule increases electron density on the nitrogen atom of the  $>C=N$  group. This leads to the strong adsorption of such molecule on the metal surface thereby resulting in high inhibition efficiency.
2. The results also suggests that the chemisorption and physisorption have simultaneous effect on the inhibition efficiency of the studied Schiff Base
3. Amongst the two inhibitors studied, *p*-AnsNB has found to be the best inhibitor.

#### ACKNOWLEDGMENT

The authors are grateful to the Chemistry Department, School of Sciences, Gujarat University, Gujarat, for laboratory facilities and also to the promoter of the journal.

# International Journal of Innovative Research in Science, Engineering and Technology

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