



EXPERIMENTAL STUDIES ON THE INHIBITION EFFECT OF ANILINE-N-(P-METHOXYBENZYLIDENE) SCHIFF BASE ON ZINC CORROSION IN HYDROCHLORIC ACID

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ABSTRACT:

The inhibition effect of Schiff base aniline-N-(p-methoxybenzylidene) (ANPMB) on the corrosion behavior of zinc in 0.5 M HCl was studied using galvanostatic polarization, electrochemical impedance spectroscopy (EIS) and weight loss method. The results showed that ANPMB is an excellent corrosion inhibitor for zinc in acidic medium. Inhibition efficiency increased with increase in the concentrations of ANPMB but decrease with rise in temperature. The adsorption of the inhibitor on the zinc surface is found to accord with Langmuir isotherm. Some thermodynamic and activation parameters have been calculated. The mechanism of inhibition is proposed.

KEY WORD: *Zinc, Corrosion inhibitor, Adsorption isotherm, Galvanostatic polarization, Thermodynamic parameters, EIS.*

INTRODUCTION:

Corrosion is defined as the deterioration of the material, usually a metal, because of reaction with its environment and which requires the presence of an anode, a cathode, an electrolyte and an electrical circuit [1]. Acid solutions are generally used for the pickling, industrial acid cleaning, acid descaling, oil well acidizing, etc [2-5]. Zinc is a metal with numerous industrial applications and is mainly used for the corrosion protection of steel [6]. Zinc is an industrially important metal and is corroded by many agents, of which aqueous acids are the most dangerous [7]. Looking at its increasing use, the study of corrosion inhibition is of paramount importance. The dissolution behavior of zinc in acidic medium is known to be inhibited by nitrogen, oxygen and sulfur-containing organic compounds. Such compounds contain electron-donating groups that would decrease the corrosion rate by increasing the hydrogen

over voltage on the corroding metal [8]. Studies of the effect of organic additives on the corrosion rate of zinc have been the subject of many investigators [9-14]. It has been found that most of the organic inhibitors act by adsorption on the metal surface [15]. This phenomenon is influenced by certain physicochemical properties of inhibitor molecule such as functional groups, steric factors, aromaticity, electron density at the donor atoms and the electronic structure of the molecule [16]. p-methoxybenzaldehyde moiety containing compounds are having potential characters of an inhibitor like the presence of hetero atoms nitrogen, oxygen and an aromatic ring together. The present study was undertaken to ascertain and compare the ability of these compounds to safeguard zinc from the corrosive action of HCl. The variation of the inhibition efficiency with temperature and concentration of the inhibitor was evaluated by mass loss method. Tafel and EIS data were also used to analyze the inhibition action. Recently, Schiff base compounds have been of interest in order to obtain efficient corrosion inhibitors since they provide much greater inhibition by adsorption on metal surface compared with corresponding amines and aldehydes [17-24]. Some Schiff's bases have recently reported as effective corrosion inhibitors for steel [25, 26], aluminium [27], aluminium alloys [28, 29], copper [30] and zinc [31] in acidic media. The presence of $-C=N-$ group in Schiff base molecules enhances their adsorption ability and corrosion inhibition efficiency [32, 33]. The action of such inhibitors depends on the specific interaction between the functional groups and the metal surface. So it is very important to clarify the interactions between the inhibitor molecules and metal surface in order to search efficient corrosion inhibitors [34].

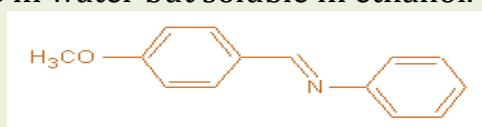
In the present work, the inhibitive effectiveness of ANPMB has been studied in retarding corrosion of zinc in 0.5 M HCl. Galvanostatic polarization, electrochemical impedance spectroscopy (EIS) and weight loss method were used to investigate the inhibiting influence of ANPMB.

MATERIALS AND METHODS:

Synthesis of Schiff base

The used Schiff base was synthesized from equimolar amounts of p-methoxybenzaldehyde and corresponding amine (aniline) through a condensation reaction in ethanol media. The Schiff base namely Aniline-N-(p-methoxybenzylidene), ANPMB, (m.p., 63°C, registry number, 836-41-9) is light yellow crystalline substance, insoluble in water but soluble in ethanol.

Chemical structure:



ANPMB

Electrodes and electrolytes

Rectangular specimens of zinc of size 3 cm × 3 cm and thickness 0.139cm with a small hole of about 2 mm diameter just near the upper end of the specimen were used for the determination of weight losses. The specimens were polished using successively '0' to '0000' Oakey emery paper. The specimens degreased by ethanol. For polarization and impedance measurements, metal coupons of circular design, diameter 2.802 cm with a handle 3 cm long and 0.5 cm wide and thickness 0.139 cm with a 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 cm² exposed to the solution. The corrosive solution (0.5 M HCl) was prepared by dilution of analytical grade 37% HCl with double distilled water. The concentration range of employed inhibitors was 0.0001 to 0.5% in 0.5 M HCl.

Measurements

Three methods i.e. polarization study, impedance spectroscopy and weight loss method were used to determine the corrosion inhibition efficiencies of Schiff base.

Electrochemical measurements

Electrochemical experiments were carried out using a standard electrochemical three-electrode cell. Electrolytic zinc was used as working electrode, platinum as counter electrode and saturated calomel electrode (SCE) as reference electrode. The test solution was contained in a H-type (80 ml in each limb) Pyrex glass cell with Luggin capillary as near to the electrode surface as possible and a porous partition to separate the two compartments. 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 in polarization method. In this study, the current density was varied in the range of 1.62×10^{-4} to 3.25×10^{-2} A cm⁻². The inhibition efficiencies at different inhibitor concentrations are calculated using the following equation:

$$(\%IE) = \frac{I^{\circ}_{\text{corr}} - I_{\text{corr}}}{I^{\circ}_{\text{corr}}} \times 10 \quad (1)$$

Where, I°_{corr} and I_{corr} are the corrosion current densities in the absence and presence of the inhibitor respectively.

Electrochemical impedance measurement was carried out in the frequency range of 30 kHz – 1 Hz at the open circuit potential, after 1/2 hour (30 minutes) of immersion, by applying 0.5 mV sine wave ac

signal using Autolab PGSTAT 302N. Double layer capacitance (C_{dl}) and charge transfer resistance (R_{ct}) values were calculated from Nyquist plots as described by Hosseini [31]. EIS data was analyzed using NOVA 1.9.16 software. The inhibition efficiencies at different inhibitor concentrations were calculated using the following equation:

$$(\%IE) = \frac{R_{ct} - R_{ct}^0}{R_{ct}} \times 100 \quad \dots\dots\dots (2)$$

where R_{ct} and R_{ct}^0 are the charge transfer resistance of the electrode with and without the inhibitor respectively.

Weight loss (chemical) method

In weight loss method, the specimens were exposed to 0.5 M HCl solution containing controlled addition of ANPMB in the range 0.0001% to 0.5% inhibitor concentration. Only one specimen was suspended by a glass hook in each beaker containing 230 ml of the test solution which was open to the air at $35^\circ \pm 0.5^\circ\text{C}$, to the same depth of about 1.5cm below the surface of the test solution. The experiments were repeated at different temperatures, ranging from $35^\circ \pm 0.5^\circ\text{C}$ to $65^\circ \pm 0.5^\circ\text{C}$ in the absence and presence of 0.1 % and 0.5% inhibitor's concentration after 30 minutes.

The percentage inhibition efficiency (%IE) was calculated using the following equation:

$$\text{Inhibitor efficiency } (\%IE) = \frac{W_u - W_i}{W_u} \times 100 \quad \dots\dots\dots (3)$$

$$\theta = \frac{W_u - W_i}{W_u} \quad \dots\dots\dots (4)$$

where, W_u = weight loss of zinc in uninhibited acid and W_i = weight loss of zinc in inhibited acid, respectively.

RESULTS AND DISCUSSION:

Polarization study

The galvanostatic polarization curves of zinc in 0.5 M HCl in the presence and absence of two different concentrations of ANPMB is shown in Fig.1.

From the Fig.1, it was observed that in the presence of ANPMB, both of the cathodic and anodic curves show low current density than those observed in the uninhibited solution [without the inhibitor]. This behavior indicated that the Schiff base has effect on both cathodic and anodic reactions of corrosion process. Therefore, this Schiff base could be classified as mixed type inhibitor with a predominantly cathodic one. ANPMB shift the potential in the negative direction, this effect is more evident at higher

concentrations, which means that the inhibitor affect cathodic reaction more than anodic reaction. The values of cathodic Tafel slopes (b_c) were calculated from the linear region of the polarization curves are given in Table-1.

The corrosion current density (I_{corr}) was determined from the intersection of the linear parts of the cathodic curves with stationary corrosion potential (E_{corr}).

The Tafel parameters and inhibition efficiencies (%IE) calculated from the polarization data are given in Table-1. The inhibition efficiencies obtained by extrapolation of the cathodic Tafel line to the corrosion potential agree well with those calculated from weight loss data. This suggests that in presence of ANPMB, both the cathode and the anode are polarized, but the cathode is polarized to a greater extent. The inhibitor thus appears to be mixed type inhibitors with predominant action on the cathode.

Electrochemical impedance spectroscopy

The impedance measurement was carried out after immersion of 60 minutes in 0.5 M HCl solution in absence and presence of different concentrations of ANPMB. The Nyquist plot for various concentrations of Schiff base is given in Fig.2.

As we can see from Fig.2, the impedance diagrams show semi-circle indicating a barrier layer formed on zinc metal surface. R_{ct} increases with increase in inhibitor concentration indicating that charge transfer reactions occurring at the protective film are strongly restricted by the adsorbed inhibitor molecules [3]. The equivalent circuit model used to fit the experimental results is shown in Fig.3. The change in concentration of inhibitor did not change the profile of the impedance behavior. This suggests that mechanism of inhibition of zinc does not change with change in concentration.

The NOVA 1.9.16 software is used for impedance data analysis and the fit parameters are listed in Table-2. Where, R_{ct} , R_s and C_{dl} are the charge transfer resistance, solution resistance and double layer capacitance, respectively.

The data of Table-2 showed that the R_s values are very small compared to the R_{ct} values. It should be noted that while R_{ct} values increase, the capacitance C_{dl} values decrease.

The double layer between the charged metal surface and the solution is considered as an electrical capacitor.

The adsorption of ANPMB on the electrode surface decreases its electrical capacity as they displace the water molecules and other ions originally adsorbed on the surface [3, 33]. The R_{ct} values increased with inhibitor concentrations may suggest the formation of a protection layer on the electrode surface.

Weight loss measurements

Effect of inhibitor concentration :

To study the effect of inhibitor concentration on inhibition efficiency, weight losses were determined in 0.5 M HCl plain as well as inhibited, at $35^{\circ} \pm 0.5^{\circ}\text{C}$ for an immersion time of 30 minutes. Table-3 shows the calculated values of inhibition efficiencies (%IE) using the expression given in eq. (3).

Increasing the concentration of ANPMB, increase the inhibition efficiency (%IE) up to a maximum value of 99.9 % at 0.5 % inhibitor concentration.

The inhibition was found to depend on inhibitor's concentration. The inhibitory action of ANPMB against Electrolytic zinc corrosion can be attributed to the adsorption of its molecules on zinc surface, which limits the dissolution of the latter by blocking its corrosion sites and hence decreasing the weight loss, with increasing efficiency as the concentration increases.

Adsorption isotherm

The inhibition efficiency depends on the type and number of active sites at the metal surface, the charge density, the molecular size of the inhibitor, the metal-inhibitor interaction, and the metallic complex formation [34]. The adsorption isotherm can give information on the metal-inhibitor interaction. The type of adsorption of ANPMB Schiff base was elucidated from the degree of surface coverage (θ) values calculated from eq. (4) (Table-3). The values of surface coverage, θ for the inhibitor have been used to explain the best isotherm which determines the adsorption process. Attempts were made to fit θ values to various adsorption isotherms namely Langmuir, Freundlich and Temkin. By far the best fits were obtained with Langmuir adsorption isotherm.

When plots of C/θ versus C (inhibitor concentration) were drawn (Fig.4), straight line was obtained, indicating that the adsorption of ANPMB takes place following Langmuir isotherm [35].

Effect of temperature

To study the effect of temperature on the inhibition efficiency, weight losses were determined at 35°C , 45°C , 55°C and 65°C in 0.5 M HCl, plain as well as inhibited. The results given in Table-4 show that the inhibition efficiencies decrease with rise in temperature.

The decrease in inhibition efficiencies with increase in temperature can be explained by desorption of the inhibitor molecules due to a higher rate of hydrogen evolution at elevated temperatures which may disturb or detach the adsorbed film from the metal surface.

Thermodynamic parameters

The thermodynamic parameters derived from the temperature data for the studied Schiff base is given in Table-5.

Activation energies for zinc dissolution process can be evaluated from the following relationship [36] :

$$\rho = k \exp \left(- \frac{E_a}{RT} \right) \dots\dots\dots (5)$$

where ρ is the corrosion rate, E_a is the activation energy, T is the thermodynamic temperature and k is constant. Plotting $\log \rho$ versus $T^{-1} \times 10^4$ gave a straight line with slope of $-E_a / 2.303 R$ (Fig.5).

Calculated activation energies are given in Table-5. As we can see from these data, activation energies strongly increase in the presence of ANPMB. The presence of inhibitor, which results in the blocking of the active sites, must be associated with an increase in activation energy E_a , which indicates the physical adsorption or weak chemical bonding between the inhibitor molecules and zinc surface [37].

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, θ , of the metal surface in a uniform random manner then the heat of adsorption, (Q_{ads}), of the inhibitors can be calculated from the equation:

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

The values of the free energy of adsorption (ΔG_{ads}) were calculated from the following equation [35]:

$$\log C_{inh} = \log \frac{\theta}{1-\theta} - \log B \dots\dots\dots (7)$$

where $\log B = - 1.74 - \left(\frac{\Delta G_{ads}}{2.303RT} \right) \dots\dots\dots(8)$

The values of (ΔG_{ads}) and (Q_{ads}) are shown in Table-5. The negative value of (ΔG_{ads}) ensures the spontaneity of adsorption process and stability of the adsorbed layer on zinc surface. Generally the value of (ΔG_{ads}) around -20 kJ mol^{-1} or lower are consistent with physisorption, while those around -40 kJ mol^{-1} or higher value involve chemisorption [38]. The values of (ΔG_{ads}) for ANPMB indicate that the molecules are physisorbed at low concentration and chemisorbed at higher concentration. The negative values of (Q_{ads}) indicated that the adsorption of used inhibitor on zinc surface is exothermic.

Mechanism of the inhibition

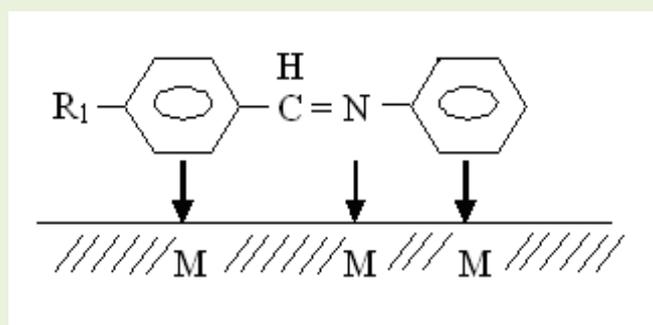
Most organic corrosion inhibitors are compounds with at least one polar unit containing atoms of nitrogen, sulfur, oxygen and in some cases selenium and phosphorous. The polar unit is regarded as the reaction centre for the establishment of the adsorption process and the adsorption bond strength is determined by the electron density on the atom acting as the reaction centre and by the polarisability of the fundamental unit. According to Hackerman [39], amine-type inhibitors have electron-donating ability and their action is attributed to the adsorption of the molecule on the metal surface through an unshared pair of electrons belonging to the nitrogen atom. The inhibitor molecules are adsorbed

chemically on the surface of the bulk metal, M, forming a charge transfer complex between the polar atom/atoms and the metal,



Further, these inhibitors, which are in a distinct ionic form, may also get attached to the metal surface of opposite polarity through electrostatic attraction. The adsorbed layer then will block the dissolution of the metal. The size, orientation and shape of the molecule, and the electronic charge on it, will determine the degree of adsorption and hence the effectiveness of the inhibitor.

It is also possible that the compounds may form onium ions in acidic medium and move to the cathodic regions and then the adsorption will take place through the iminic nitrogen and also through the -OCH₃ group (+R, -I) which supplies electron density to the aromatic benzaldehydic ring and thus the delocalized π-electrons of this ring help in the adsorption of the compound with stronger bonds. It appears that the inhibitor molecules lie flat on the metal surface and cover greater area when present in sufficient amount.



Where, R₁ = -OCH₃ in ANPMB.

CONCLUSION:

The inhibition of zinc in 0.5 M HCl with and without different concentrations of ANPMB has been investigated by polarization method, EIS and weight loss method and the main conclusions are:

- (1) ANPMB is good inhibitor in 0.5 M HCl solutions and the inhibition efficiency up to 99.9% was obtained. The inhibition efficiencies of inhibitor increased with the increase of inhibitor concentration.
- (2) Adsorption of ANPMB in 0.5 M HCl solution on zinc surface was found to obey the Langmuir adsorption isotherm.
- (3) ANPMB act as mixed type inhibitor with predominant action on cathode.

(4) The adsorption takes place due to the iminic group and $-OCH_3$ group, therefore the inhibitive efficiency of the Schiff base should be almost same.

Obtained results about inhibition efficiencies from polarization study, EIS and weight loss method are in good agreement with each other.

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Table-1 Electrochemical parameters of corrosion of zinc in the presence of different concentration of inhibitors at 35°C ± 0.5°C and corresponding inhibition efficiencies obtained from polarization method.

Inhibitor	Concentration (% W/V)	E _{corr} (mV)	b _c (mV/dec)	I _{corr} for cathodic (A cm ⁻²)	(%IE)
Blank	-	-1019	169	4.17*10 ⁻³	-
ANPMB	0.1	-945	151	1.15 *10 ⁻³	72.42
	0.5	-930	166	6.45*10 ⁻⁸	99.99

Table-2 Impedance parameters and corresponding inhibition efficiency for the corrosion of zinc in the 0.5 M HCl.

Inhibitor	Concentration (% W/V)	R _s (ohm)	R _{ct} (ohm)	C _{dl} (μF)	IE (%)
Blank	-	0.688	5.99	183	
ANPMB	0.1	2.23	37.9	6.18	84.2
	0.15	2.12	89.4	3.40	93.3
	0.2	2.11	106	3.14	94.4

Table-3 Corrosion parameters for zinc in the presence and absence of different concentrations of Schiff base obtained from weight loss measurements at 35°C ± 0.5°C for exposure period of 30 minutes.

Inhibitor	Concentration (% W/V)	Weight loss (mg dm ⁻²)	(%IE)
Blank	-	7930	-
ANPMB	0.1	1803	77.26
	0.15	71	99.10
	0.2	46	99.42
	0.3	20	99.74
	0.4	10	99.87
	0.5	5	99.94

Table-4 Effect of temperature on weight loss and inhibition efficiency for zinc in 0.5 M hydrochloric acid.

Inhibitor	Concentration (% w/v)	Weight loss (mg dm ⁻²) at temperature			
		35 °C	45 °C	55 °C	65 °C
Blank	-	7939	9439	13553	16017
	0.1	1803	3825	8463	10317
ANPMB	0.5	5	7	13	16

Table-5 Thermodynamic parameters and activation energy for inhibitor adsorption for corrosion of zinc in 0.5 M HCl (inhibitor concentration 0.5%).

Inhibitor	E _a (kJ mol ⁻¹)	Q _{ads} (kJ mol ⁻¹)	ΔG _{ads} (kJ mol ⁻¹)
Blank	10.2	-	-
ANPMB	41.0	-54.8	-39.3

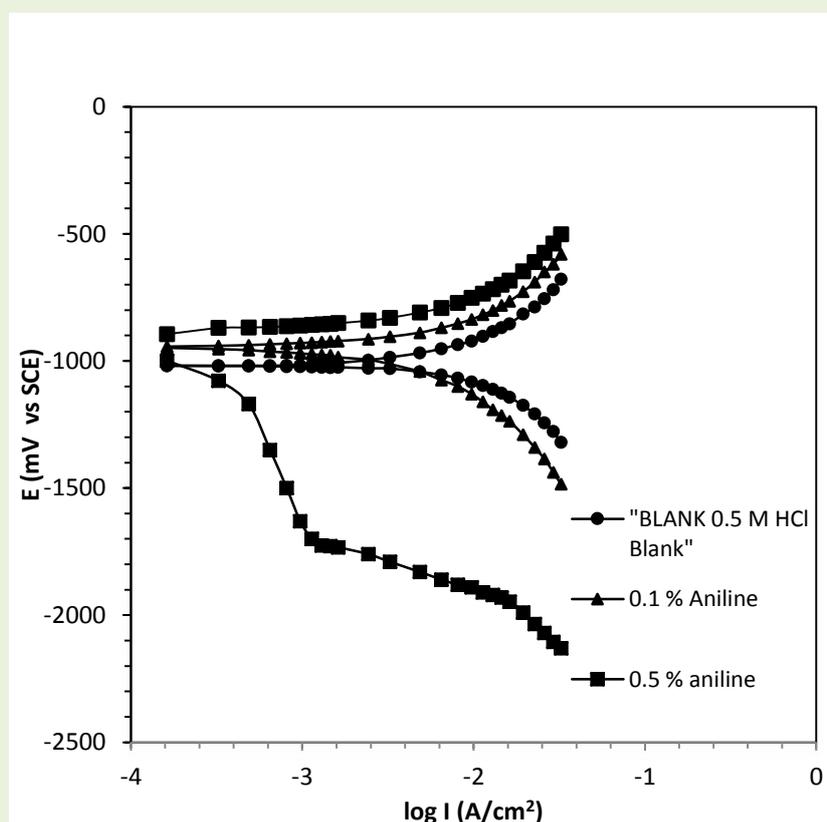


Fig. 1: Anodic and cathodic polarization curves obtained for zinc metal at 35°C ± 0.5°C in 0.5 M HCl in various concentration of studied Schiff base.

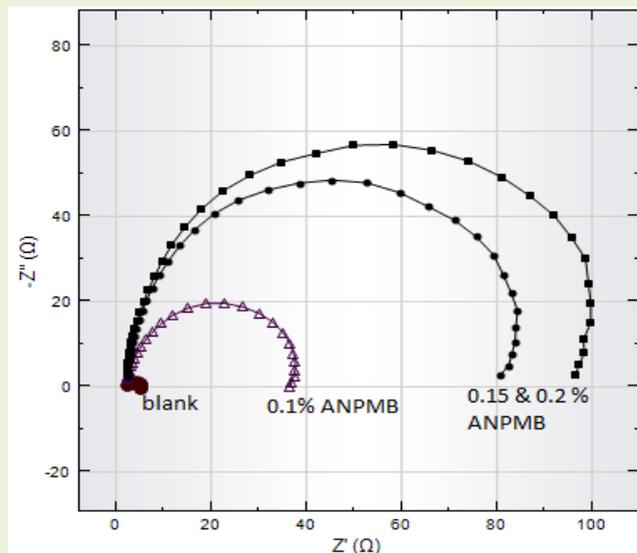


Fig. 2: Impedance plot obtained at 35°C in 0.5 M HCl in various concentration of Schiff base.

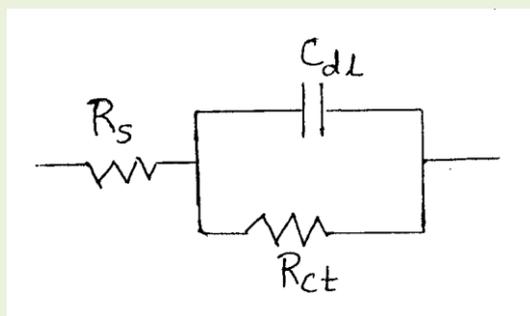


Fig. 3: The equivalent circuit model used to fit the experimental results.

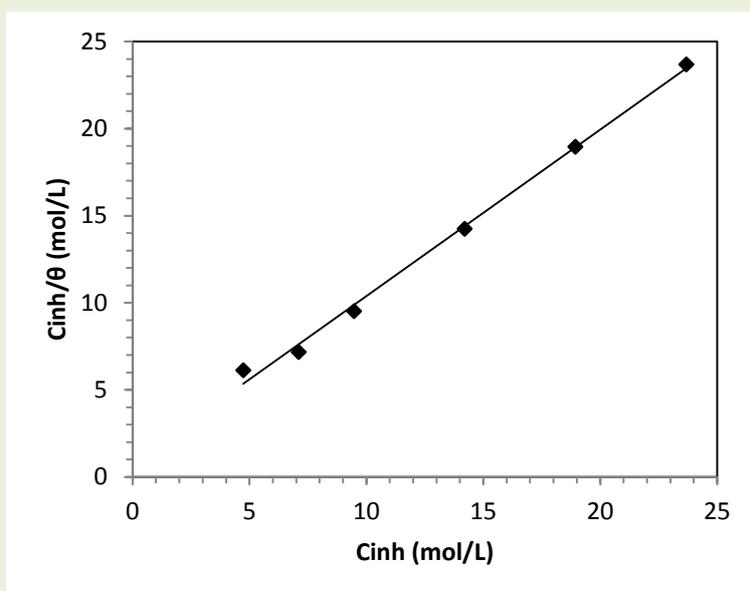


Fig. 4: Langmuir isotherm for adsorption of ANPMB on the zinc metal surface.

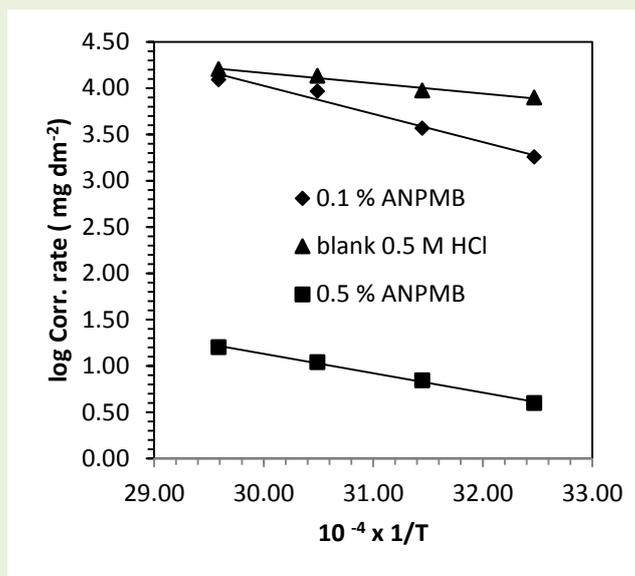


Fig. 5: Plotting $\log \rho$ vs. $T^{-1} \times 10^4$ to calculate the activation energy of corrosion process in the presence and absence of ANPMB.