

INHIBITION OF PITTING CORROSION OF SOME AUSTENITIC STAINLESS STEEL SAMPLES IN 2.5 M NaCl SOLUTION BY 3-AMINO-1,2,4-TRIAZOLE

ABSTRACT

The effect of addition of 3-AMINO-1,2,4-TRIAZOLE (ATA) on general and pitting corrosion of three Egyptian austenitic stainless steel samples, namely SS 304L (0.09 Mo), SS 304H (0.43 Mo) and SS 304H (1.15 Mo) in aerated 2.5 M NaCl solution was investigated. The measurements were carried out using potentiodynamic polarization and the Tafel lines methods. The influence of temperature on the inhibition properties of ATA was investigated. The results indicate that ATA is a good inhibitor for the samples under investigation in 2.5 M NaCl solutions. In addition, the inhibition efficiency $\eta\%$ increases with the inhibitor concentration while it decreases with increasing temperature. Moreover, addition of ATA to 2.5 M NaCl solution leads to a shift of the pitting potential towards the noble direction accounting for increased resistance to pitting attack. The inhibitive properties of the ATA were explained on the bases of its adsorption on the stainless steel samples and formation of adsorbed barrier layer, which resist the penetration power of Cl^- ion and consequently decreases the rate of pitting corrosion. The adsorption of the inhibitor obeys a Temkin adsorption isotherm. Examination of polarization curves indicates that ATA acts as a mixed-type inhibitor.

Keywords: Pitting corrosion, Stainless steel, 3-amino-1,2,4-triazole, Tafel lines, Temkin.

1. INTRODUCTION

The austenitic stainless steel is the most common and important type in the stainless steel family which are characterized by high corrosion resistance [1]. Austenitic stainless steel has excellent ductility, formability and toughness, even at cryogenic temperatures. They are widely used as a construction material in various aggressive environments such as piping systems, heat exchanger equipment, drilling platforms, chemical plants, desalination plants, and wastewater treatment plants. In addition, most of the containers and food contact equipments are manufactured from the austenitic grades of stainless steel [2]. The high corrosion resistance of the stainless steel is attributable to the presence of passive film [3]. This film is stable, invisible, thin, durable and extremely adherent and self repairing. The stability of passive films and the reactions taking place in the transpassive potential region depend on the nature of the anode potential and the anion present. Although

austenitic stainless steels show a very high corrosion resistance in many aggressive environments, they can suffer pitting corrosion in chloride environments [4-6]. It is well known that Cl^- ions are aggressive enough to attack steel and initiate pitting [7–10]. The use of inhibitor is one of the most practical methods for protection of the metal against the corrosion but few compounds can be applied successively [11,12]. The mechanism of corrosion inhibition by organic molecules is mostly attributed due to their adsorption onto metallic surfaces. The adsorption phenomenon depends, principally, on the chemical composition and structure of the inhibitors, nature of the metallic surface, and the properties of the medium where the inhibitor-surface interaction takes place [13]. Compounds containing hetero-atoms such as sulphur, nitrogen and oxygen containing compounds exhibit the most efficient corrosion inhibition of metals. Survey of literature reveals that triazole and its derivatives are effective corrosion inhibitors for different metals and alloys [14-18].

Therefore, the present work aims to test the applicability of ATA as a pitting corrosion inhibitor for SS 304L (0.09 Mo), SS 304H (0.43 Mo) and SS 304H (1.15 Mo) samples in 2.5 M NaCl. It is also aimed to predict the thermodynamic feasibility of adsorption of the inhibitor molecule on the stainless steel surface and to study its adsorption behavior.

2. EXPERIMENTAL DETAILS

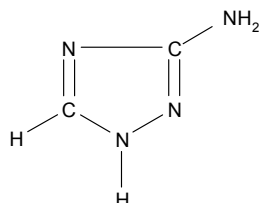
The austenitic stainless steel samples used in the present work were produced in the induction furnace of the Modern Foundries Company, Giza, Egypt. The working electrode was in the form of rod (5 mm diameter) and was enclosed in a Pyrex glass tube sealed with Araldite to offer an exposed surface area of 0.28 cm^2 . The chemical composition of these alloys and their abbreviations are given in Table 1.

Table 1: Chemical composition (wt %) of the austenitic stainless steel alloys

Alloy symbol	Alloy	C	Si	Mn	Cr	Ni	Mo	P	S	Cu
I	SS 304L (0.09 Mo)	0.033	0.330	1.900	18.01	8.11	0.09	0.031	0.004	2.180
II	SS 304H (0.43 Mo)	0.200	1.300	1.300	17.75	8.48	0.43	0.026	0.014	0.200
III	SS 304H (1.15 Mo)	0.220	1.250	1.200	19.45	9.45	1.15	0.025	0.015	0.200

The electrode was polished with a series of emery papers, from a coarse one 500 and proceeding in steps to fine grade 1500, washed thoroughly with doubly distilled water, and then introduced into the test solution. A platinum wire was used as a counter electrode. All potentials were measured against a saturated calomel electrode. The measurements were carried out in three-electrode cell by using a computer-assisted Potentiostat ACM. The cell was placed in a water thermostat to attain the required temperature $\pm 1.0^\circ\text{C}$. All solutions were freshly prepared from analytical grade chemicals and doubly

distilled water. The aqueous corrosion behavior of the samples was studied by using potentiodynamic polarization technique. The potentiodynamic polarization curves were recorded by sweeping the potential from the starting potential towards more positive direction with scan rate of 20 mVs⁻¹. In all cases, duplicate experiments were carried out to ensure reproducibility. The 3 - amino-1,2,4-triazole additive used was purchased as chemically pure grade (BDH) and it has the following structure:



3.RESULTS AND DISCUSSION

3.1. Potentiodynamic polarization curves and Tafel lines

Figs. 1a- 3a demonstrate the cathodic and anodic potentiodynamic polarization curves of the austenitic stainless steel samples I, II and III respectively in 2.5 M NaCl solution without and with various concentrations of ATA at 25° C. Around the zero current potential, the polarization curves exhibit Tafel behavior. The corrosion current, j_{corr} , and the corrosion potential, E_{corr} , were calculated by extrapolation of the linear logarithmic sections of the cathodic and anodic Tafel lines to the point of intersection (Figs.1b-3b). The potentiodynamic polarization curves were swept starting from -1800 mV towards more positive directions at a scan rate of 20 mVs⁻¹. Inspection of these curves reveals that the cathodic current density of each sample decreases as the potential moves positively. It is also clear that the potentiodynamic polarization curves are characterized by a very wide potential domain of passivity especially for alloy III (≈ 2500 mV) (Fig. 3a). In addition, the curves do not exhibit active-passive transition. Moreover, during the anodic scan, the passive currents are very small indicating the good protection efficiency of the samples not only on the corrosion potential domain but also in the passive potential domain.

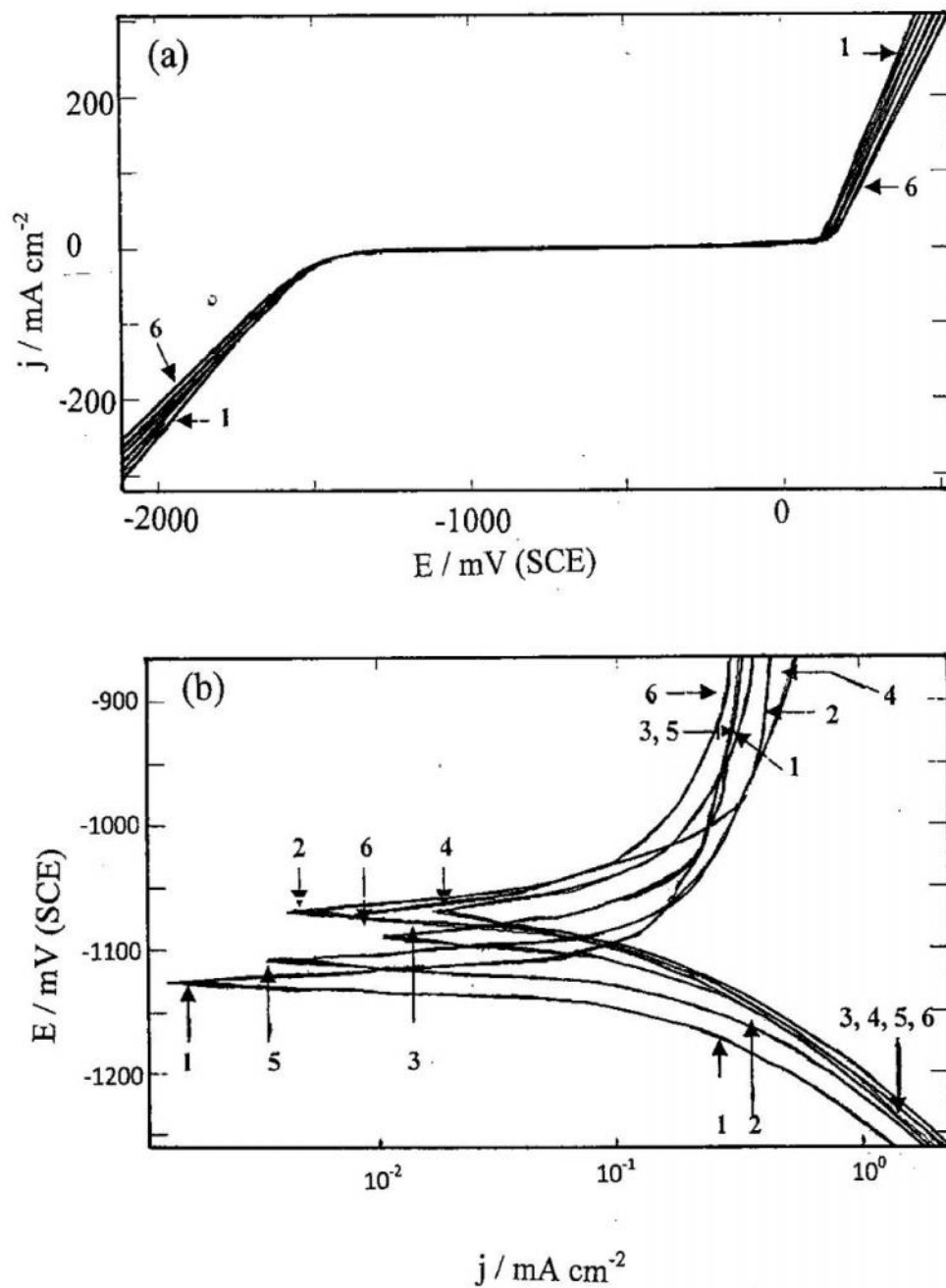


Fig. 1 (a) Potentiodynamic cathodic and anodic polarization curves recorded for Alloy I (SS 304L) in 2.5 M NaCl solution in the absence and presence of various concentrations of ATA at a scan rate of 20 mV s^{-1} and at 25°C (1) Blank; (2) 10^{-7} M ; (3) 10^{-6} M ; (4) 10^{-5} M ; (5) 10^{-4} M and (6) 10^{-3} M and Fig. 1(b) Tafel plots.

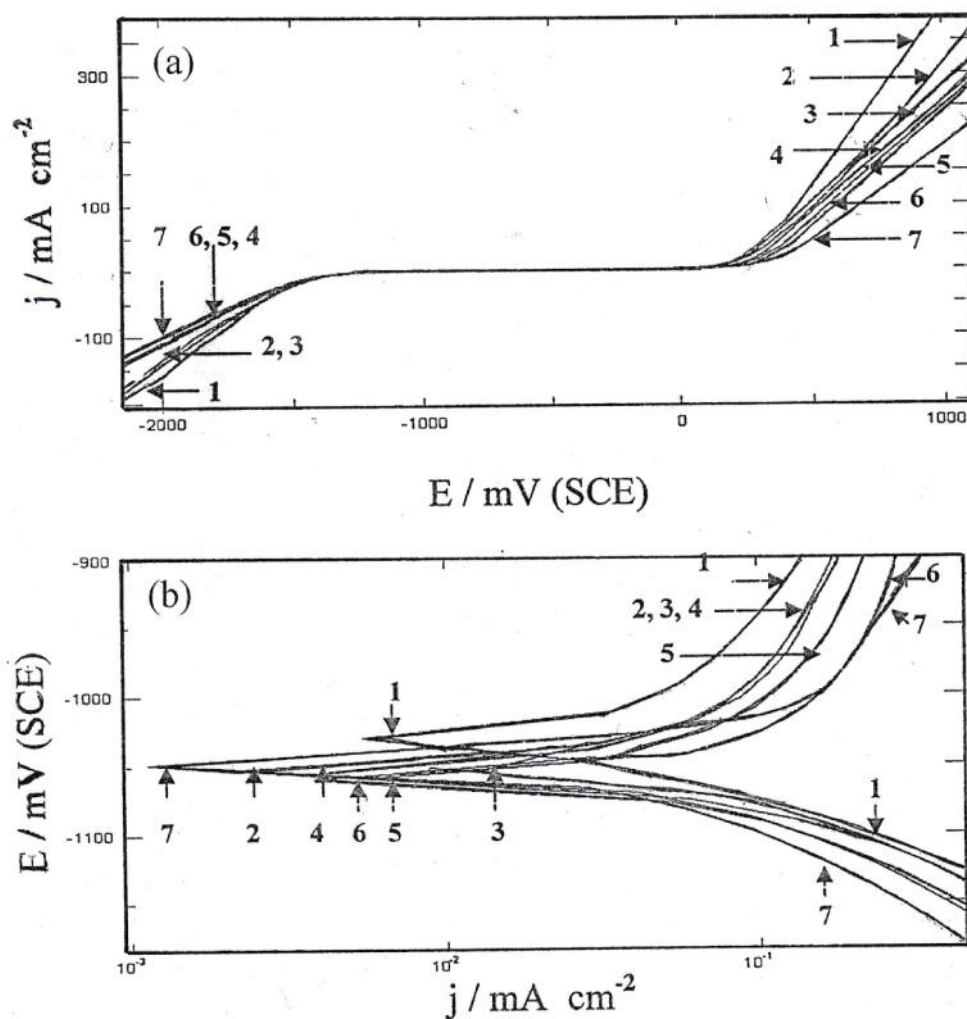


Fig. 2 (a) Potentiodynamic anodic polarization curves recorded for Alloy II (SS 304H) (0.43 Mo) in 2.5 M NaCl solution in the absence and presence of various concentrations of ATA at a scan rate of 20 mVs^{-1} and at 25°C (1) Blank; (2) 10^{-8} M ; (3) 10^{-7} M ; (4) 10^{-6} M ; (5) 10^{-5} M ; (6) 10^{-4} M and (7) 10^{-2} M and Fig. 2 (b) Tafel plots.

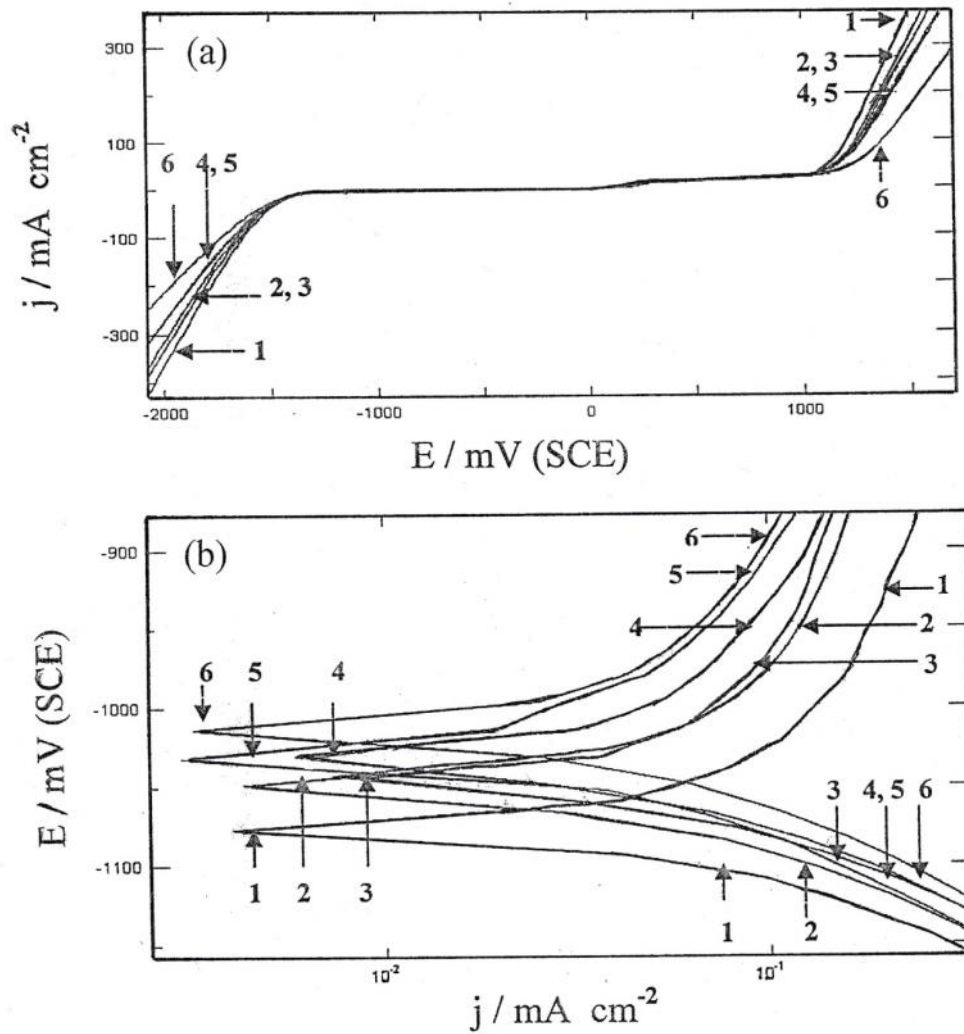


Fig. 3 (a) Potentiodynamic anodic polarization curves recorded for Alloy III (SS 304H) (1.15 Mo) in 2.5 M NaCl solution in the absence and presence of various concentrations of ATA at a scan rate of 20 mVs^{-1} and at 25°C (1) Blank; (2) 10^{-8} M ; (3) 10^{-7} M ; (4) 10^{-6} M ; (5) 10^{-5} M and (6) 10^{-2} M and **Fig. 3 (b)** Tafel plots.

Several models have been proposed for the existence of the passive film on the alloy surface of austenitic stainless steel in aqueous chloride solutions [19,20]. It is clear that the passive region in all cases extends up to a certain critical potential, pitting potential E_{pit} , at which the small passive current density increases rapidly without any sign for oxygen evolution, indicating initiation of pitting corrosion (Figs. 1a–3a). Once a pit is nucleated, pitting growth and propagation is proceeded in active dissolution mode. On the other hand, it is evident that, from the electrochemistry point of view, Mo can dissolve at relatively low potentials due to formation of soluble MoO_4^{2-} species [21]. The potential region of this reaction is situated in the passive region of Fe base alloys. Hence, dissolved Mo species play a role on passivity and its breakdown.

Inspections of the data in Table 2 reveal that addition of ATA decreases the values of j_{corr} and hence the rate of corrosion of the three samples indicating that ATA acts as a corrosion inhibitor. Moreover, addition of ATA has no significant effect on E_{corr} , implying that it acts mainly as a mixed type inhibitor. The inhibition function of ATA molecules is due to their adsorption and formation of adsorbed barrier layer on the electrode surface. Adsorption of these molecules takes place via electrostatic interaction between the amino group and π electrons of hetero-ring and the alloy surface. The adsorbed ATA molecules may block the cathodic and anodic sites on the electrode surface. Such process inhibits both cathodic reduction of oxygen and anodic dissolution of alloys and enhances the cathodic and anodic overpotentials. An increase in the concentration of ATA decreases the value of j_{corr} as a result of increasing the surface coverage and decreasing the surface area available for corrosion reactions. The values of β_c and β_a ($\beta_c = 791$ and $\beta_a = 707$ mV decade⁻¹) remain almost unchanged with the variation of ATA concentrations. Such behavior demonstrates that the presence of ATA in the solution has no effect on the mechanism of the dissolution process of the samples, but the adsorbed inhibitor molecules mechanically screen the coated part of the electrode and, therefore, protect it from the action of the corrosion medium.

The percent of inhibition efficiency (η %) of ATA was calculated in all cases using the following equation:

$$\eta\% = 100 \left(1 - \frac{j_{corr}}{j_{corr}^0} \right) \quad (1)$$

where j_{corr}^0 and j_{corr} are the corrosion current density in the absence and presence of ATA in chloride solution respectively, where j_{corr}^0 and j_{corr} are the corrosion current density in the absence and presence of ATA in chloride solution respectively.

The calculated values of η are given in Table 2. It is clear that the inhibition efficiency, η , of ATA increases with an increase in its concentration for the three samples. The inhibition effect of ATA on the three samples studied follows the following order: alloy III > alloy II > alloy I. This could be explained by the fact that increasing Mo content increased passivation behavior of the stainless steel and increases resistance to pitting corrosion [22].

Table 2. Electrochemical parameters E_{corr} , j_{corr} and η % associated with polarization measurements recorded for alloy I, alloy II and alloy III in 2.5 M NaCl solution in the absence and presence of various concentrations of ATA at 25° C.

[ATA] / M	Alloy I			[ATA] / M	Alloy II			[ATA] / M	Alloy III		
	$-E_{\text{corr}}$ (mV)	j_{corr} (mA cm ⁻²)	η %		$-E_{\text{corr}}$ (mV)	j_{corr} (mA cm ⁻²)	η %		$-E_{\text{corr}}$ (mV)	j_{corr} (mA cm ⁻²)	η %
0.0	1118	68.90	-	0.0	1020	45.50	-	0.0	1080	20.85	
10^{-7}	1070	68.88	00.03	10^{-8}	1055	35.14	22.80	10^{-8}	1060	14.97	28.20
10^{-6}	1090	64.42	12.31	10^{-7}	1053	33.10	27.25	10^{-7}	1050	14.13	32.23
10^{-5}	1070	54.80	20.46	10^{-6}	1053	30.97	31.93	10^{-6}	1032	12.90	38.12
10^{-4}	1110	52.96	23.13	10^{-5}	1058	27.70	39.12	10^{-5}	1032	11.70	43.90
10^{-3}	1070	51.11	25.82	10^{-4}	1059	22.80	49.90	10^{-2}	1025	7.97	61.77
				10^{-2}	1050	19.88	56.30				

The effect of temperature (25–75°C) on the potentiodynamic polarization curves for sample I in (2.5 M NaCl + 1.0×10^{-6} M ATA) solution and for samples II and III in (2.5 M NaCl + 1.0×10^{-2} M ATA) solution were carried out and the results deduced from these curves are tabulated in Table 3. The results show that both cathodic and anodic potentials decrease with an increase of temperature. In addition, increasing the temperature shows that the inhibitor has prevalent effect on the cathodic

polarization with respect to the anodic one. The value of j_{corr} increases with an increase in temperature as a result of increasing the rate of corrosion of the three samples at higher temperatures (Table 3). The inhibition efficiency, $\eta\%$, of ATA for the three samples at different temperatures was calculated and the results are shown in Table 3. It is obvious that in all cases, the inhibition efficiency decreases with elevating temperature. Such behavior can be interpreted on the basis that an increase in temperature decreases the strength of adsorption. These results confirm the suggestion that the adsorption process is a chemisorption in nature. Similar temperature effect on the $\eta\%$, was recorded using hexadecyl pyridinium bromide as an effective inhibitor for the same stainless steel samples [12].

Table 3 Electrochemical parameters, E_{corr} , j_{corr} and $\eta\%$ obtained from polarization measurements for alloy I, alloy II and alloy III in 2.5 M NaCl solution (blank) in the absence and presence of ATA at different temperatures.

Temp/ °C	Alloy I			Alloy II			Alloy III		
	Blank	blank + 10^{-6} M ATA		Blank	blank + 10^{-2} M ATA		Blank	blank + 10^{-2} M ATA	
	j_{corr} (mA cm ⁻²)	j_{corr} (mA cm ⁻²)	$\eta\%$	j_{corr} (mA cm ⁻²)	j_{corr} (mA cm ⁻²)	$\eta\%$	j_{corr} (mA cm ⁻²)	j_{corr} (mA cm ⁻²)	$\eta\%$
25	68.90	60.42	12.33	45.5	19.88	56.30	20.85	7.97	61.77
35	91.20	62.07	31.94	70.8	38.41	45.75	58.60	23.53	59.85
45	121.0	90.02	25.60	114.0	63.78	44.05	90.00	45.64	49.29
55	155.0	120.0	22.80	147.0	89.57	39.07	112.0	65.40	41.60
65	165.0	135.0	18.20	163.0	108.00	33.74	140.0	85.50	38.92
75	170.0	142.0	16.47	165.0	114.55	30.58	155.0	97.40	37.16

The apparent activation energy E_a^0 of corrosion of the three stainless steel samples was calculated in (2.5 M NaCl + 10^{-6} M ATA) solution for alloy I and in (2.5 M NaCl + 10^{-2} M ATA) solution for alloys II and III, using Arrhenius equation:

$$\log j_{\text{corr}} = - (E_a^0 / 2.303 RT) + A \quad (2)$$

where R is the universal gas constant, T is the absolute temperature and A is the Arrhenius factor. A plot of $\log j_{\text{corr}}$, obtained from the polarization curves vs. $1/T$ gave straight lines, as shown in Fig. 4. The values of E_a^0 obtained from the slopes of the straight lines, are 16.9, 24.5, and 31.1 kJ mol $^{-1}$ for alloy I, II and III respectively. It is clear that addition of ATA to the chloride solution increases the activation energy. These values of activation energies are greater than that recorded for hexadecyl pyridinium bromide as an effective inhibitors for the same samples in H $_2$ SO $_4$ solution [12].

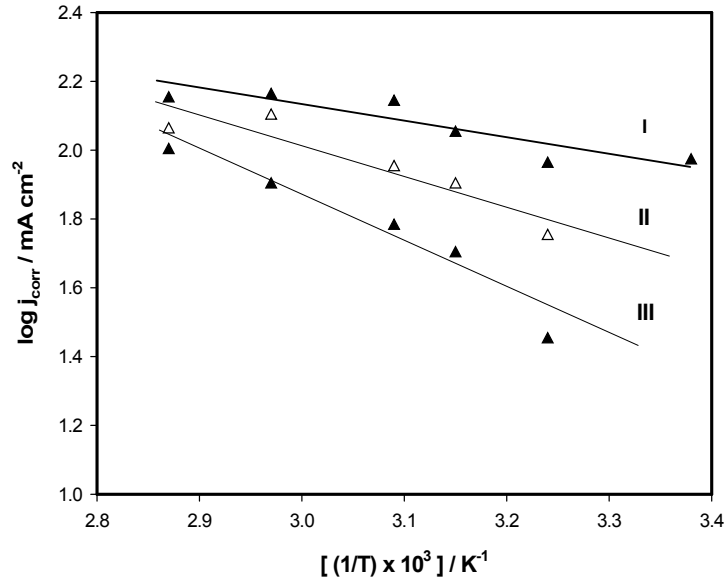


Fig. 4 Arrhenius plots of $\log j_{\text{corr}}$ vs. $(1/T)$ for alloys I, II and III in 2.5 M NaCl solution containing 10^{-6} M and 10^{-2} M ATA respectively.

3.2 Adsorption isotherm

It is found that Temkin adsorption isotherm (equation 3) fit well with the experimental data obtained from the potentiodynamic polarization measurements.

$$a \theta = \ln KC \quad (3)$$

Where θ is the fraction of the surface covered by ATA at any time ($\theta = \eta / 100$), a is the molecular interaction parameter, which depends on the lateral interaction force among the adsorbed inhibitor molecules on the surface and on the degree of heterogeneity of the alloy sample, C is the concentration of ATA in the solution and K is the equilibrium constant for adsorption process of the inhibitor. A plot of θ against $\ln C$ gave a straight line as shown in Fig. 5 with a slope of $1/a$ and an intercept of $(1/a \ln K)$. The equilibrium constant K is related to the free energy of adsorption, ΔG_{ads}^0 , by the relationship [23]:

$$\Delta G_{ads}^0 = - RT \ln (55.5K) \quad (4)$$

The value of 55.5 in the above equation is the concentration of water in the solution in mol L^{-1} . The values of K , ΔG_{ads}^0 and (a) for the three alloys are given in Table 4. The more negative value of ΔG_{ads}^0 of ATA and the increase in the value of K indicates that it is strongly adsorbed on the surface of the samples. Generally, values of ΔG_{ads}^0 up to -20 kJ mol^{-1} are consistent with electrostatic interactions between the charged molecules and the charged metal (physisorption), while those around -40 kJ mol^{-1} or higher are associated with chemisorption as a result of sharing or transfer of electrons from the inhibitor molecules to the metal surface to form a coordinate type of bond (chemisorption) [24]. The absolute values of ΔG_{ads}^0 calculated in presence of the ATA are found to be relatively high (Table 4). Therefore, the results indicate the inhibitor to be chemically adsorbed on the stainless steel surface. The data indicate also that ΔG_{ads}^0 increases for the different alloys as follows: alloy III > alloy II > alloy I.

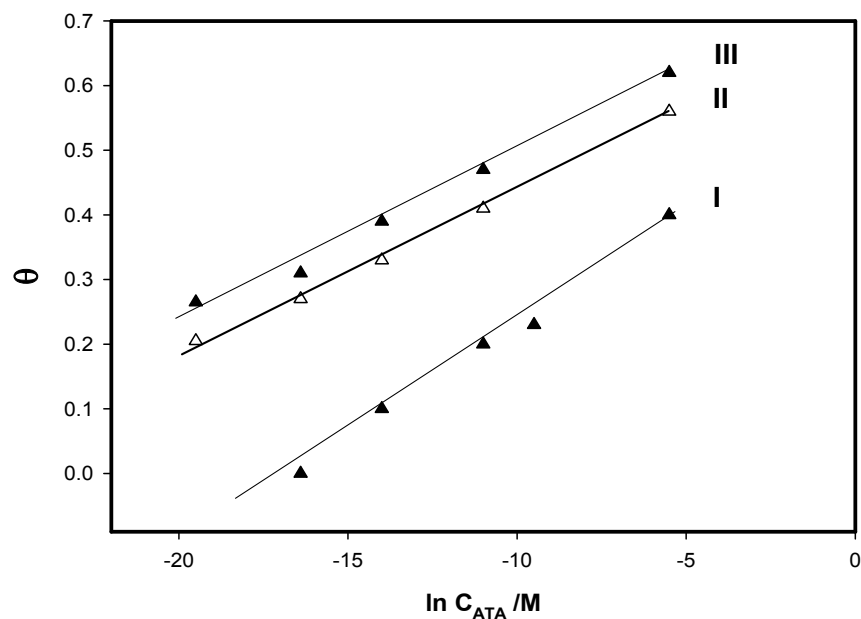


Fig. 5 Curve fitting of the data obtained from polarization measurements for alloys I, II and III in 2.5 M NaCl solution containing various concentrations of ATA to Temkin adsorption isotherm at 25°C.

Table 4 Inhibitor binding constant (k) , free energy of adsorption (ΔG_{ads}^0) and molecular interaction parameters (a) recorded for alloys I, II and III in 2.5 M NaCl solution containing various concentrations of ATA at 25°C obtained by applying Temkin adsorption isotherm.

Alloy	K	$-\Delta G_{\text{ads}}^0$ (kJ/mole)	a
I	19458291	51.50	30.03
II	6.87×10^{11}	77.48	40.45
III	6.14×10^{12}	82.91	40.45

3.3.Effect of ATA on the pitting corrosion

It is clear from Figs. 1a-3a that addition of ATA to 2.5 M NaCl solution at 25°C shifts E_{pit} to more positive values suggesting an increase in pitting corrosion resistance of the three samples I, II and III.

The values of E_{pit} increase with increasing inhibitor concentration. The relationship between E_{pit} versus $\log C_{\text{ATA}}$ is shown in Fig. 6. Linear relationships were obtained for all the samples giving an increase in the positive shift of the pitting potential as the inhibitor concentration was increased. The variation of pitting potential with inhibitor concentration obeys the following equation:

$$E_{\text{pit}} = a^* + b^* \log C_{\text{ATA}} \quad (5)$$

where a^* and b^* are constants which depend on the type of additive. The inhibition action of ATA towards pitting corrosion could be related to competitive adsorption between the aggressive Cl^- anions and ATA molecules on the surface of the passive film. The displacement of Cl^- anions by ATA molecules reduces the Cl^- surface concentration. Competitive adsorption may occur not only on the outer surface of the sample but also on the pit bottom and can reduce the rate of pit propagation.

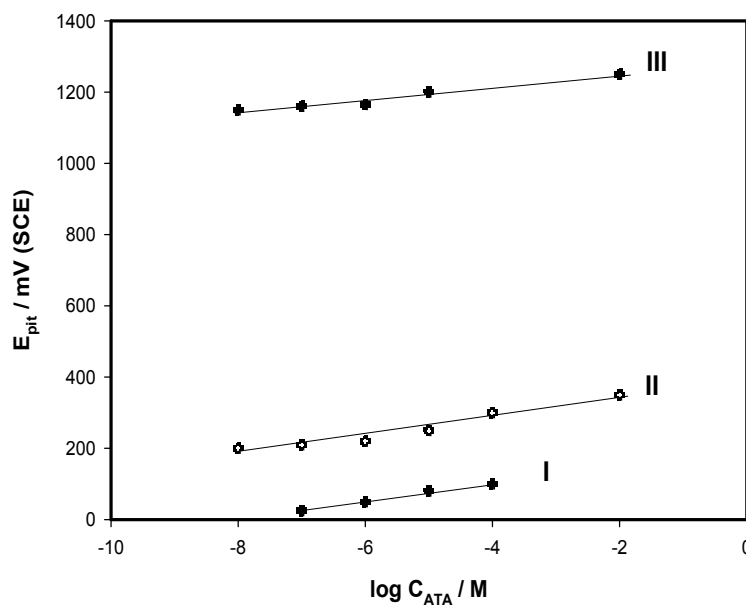


Fig. 6 Relation between pitting potential (E_{pit}) and $\log C_{\text{ATA}}$ for alloys I, II and III in 2.5 M NaCl solution at 25°C.

The effect of temperature on the values of E_{pit} for alloy I in (2.5 M NaCl + 10^{-6} M ATA) solution and for alloys II and III in (2.5 M NaCl + 10^{-2} M ATA) solution, was investigated. As expected, the values of E_{pit} decrease with raising the solution temperature. By comparing the values of E_{pit} obtained in the

absence and presence of ATA (Table 5) it is clear that the presence of ATA inhibits pitting corrosion at all temperatures studied.

Table 5 Comparison between pitting potential (E_{pit}) for alloy I in 2.5 M NaCl and in (2.5 M NaCl + 10^{-6} M ATA) solution and for alloys II and III in 2.5 M NaCl and in (2.5 M NaCl + 10^{-2} M ATA) solution at different temperatures

Temp./ °C	Alloy I		Alloy II		Alloy III	
	E_{pit} (mV)		E_{pit} (mV)		E_{pit} (mV)	
	2.5 M NaCl	2.5M NaCl + 10^{-6} M ATA	2.5 M NaCl	2.5M NaCl + 10^{-2} M ATA	2.5 M NaCl	2.5M NaCl + 10^{-2} M ATA
25	100.0	110.52	150.0	300.0	1150	1210
35	30.46	52.50	58.9	163.5	129.5	143.2
45	-25.0	-7.05	-2.29	75.0	46.5	60.7
55	-60.0	-50.27	-54.79	25.00	20.0	39.51
65	-90.73	-80.00	-56.29	-22.78	-26.5	-19.3
75	-107.0	-100.0	-116.29	-29.24	-36.54	-30.02

4.CONCLUSIONS

From the analysis of the results it was concluded that ATA is a good inhibitor for the samples under investigation in 2.5 M NaCl solutions. The inhibition efficiency η % increases with the inhibitor concentration while it decreases with increasing temperature. The inhibition effect of ATA on the three samples studied follows the following order: alloy III > alloy II > alloy I. The adsorption of the inhibitor obeys a Temkin adsorption isotherm. Addition of ATA to 2.5 M NaCl solution leads to a shift of the pitting potential towards the noble direction accounting for increased resistance to pitting attack. Examination of polarization curves indicates that ATA acts as a mixed-type inhibitor. Addition of ATA to the chloride solution increases the activation energy and the free energy change ΔG_{ads}^0

increases in the following order: alloy III > alloy II > alloy I. The values obtained for the activation energy and $\Delta G_{\text{ads}}^{\circ}$ confirmed that ATA is chemically adsorbed on the stainless steel samples.

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