

## Corrosion of High Strength Steel in Concentrated Sulfuric Acid Pickling Solutions and Its Inhibition by 3-Amino-5-mercapto-1,2,3-triazole

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The corrosion of high strength steel after varied exposure periods of time, namely 30, 90 and 180 min immersion in 2.0 M H<sub>2</sub>SO<sub>4</sub> pickling solution and its inhibition by 3-amino-5-mercapto-1,2,3-triazole (AMTA) were investigated. The study has been carried out using potentiodynamic polarization, electrochemical impedance spectroscopy and complemented by scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analyzer. It has been found that the increase of exposure period remarkably the corrosion of steel through increasing the anodic current, cathodic current, corrosion current density and corrosion rate. This effect also decreased both the solution and polarization resistances for the steel in the sulfuric acid solution. The presence of 1x10<sup>-3</sup> M AMTA and the increase of its concentration to 5x10<sup>-3</sup> M highly decreased the corrosion of the steel via the adsorption of AMTA molecules on its surface precluding its dissolution as was confirmed by the SEM micrographs and EDX profile analyses. Results together confirmed that the corrosion of steel increases with the increase of time and AMTA is a powerful corrosion inhibitor for the high strength steel in the 2.0 M H<sub>2</sub>SO<sub>4</sub> pickling solutions.

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**Keywords:** 3-amino-5-mercapto-1,2,3-triazole; acidic pickling solutions; organic corrosion inhibitors; high strength steel; impedance spectroscopy

### 1. INTRODUCTION

High-strength steels are well known to be used in various applications in industry. These steels have been used in sintering of components from powders, hyper-sonic wind tunnels, water jet cutting,

power generation, gun barrel, food sterilization, etc [1-3]. The high strength steels are designed to provide better mechanical properties along with high resistance to atmospheric corrosion than conventional carbon steels.

Acidic solutions have been widely used to remove scales and corrosion products, which have a negative effect on the performance of steel equipments [1,4]. In this regard, various ways of usage for performing the cleaning process, these are; acid pickling, industrial acid cleaning, acid descaling and oil well acidising [4]. Corrosion inhibitors are usually used during the cleaning process to reduce the corrosivity of the acid solution and to prevent it from attacking the surface of the structure to be protected [1,4-9]. It has been reported that inhibitors are mainly employed to control the metal dissolution as well as acid consumption [4]. Corrosion inhibitor is a chemical substance that when added in low concentration to a certain environment, it reduces the corrosion of metallic materials in that medium [10]. Organic compounds containing polar groups including nitrogen, sulfur, and oxygen, and heterocyclic compounds with polar functional groups and conjugated double bonds have been reported to be good corrosion inhibitors [1,4,11-20]. The inhibiting action of these compounds is usually attributed to their interactions with the metal via their adsorption onto the surface. It is usually believed that the adsorption of an inhibitor on a metal surface depends on the nature and the surface charge of the metal, the adsorption mode, its chemical structure, and the type of the electrolyte solution [15].

In our previous work, we reported the corrosion and corrosion inhibition of high strength low alloy (HSLA) steel after 10 min and 60 min immersion in highly concentrated sulfuric acid solutions by 3-amino-1,2,4-triazole (ATA). It was found that the increase of immersion time increases the corrosion the HSLA steel and that corrosion decreases in the presence and by the increase of ATA concentration. In this study, we investigated the effect of increasing the exposure periods from 30 min to 90 min and further to 180 min on the corrosion of high strength steel in 2.0 M H<sub>2</sub>SO<sub>4</sub> solutions. The objective of this work was also extended to report the inhibition of the corrosion of the steel using 3-amino-5-mercapto-1,2,3-triazole (AMTA) as a corrosion inhibitor. The study was carried out using potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) measurements and further complemented by using scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analyses.

## 2. EXPERIMENTAL PROCEDURES

### 2.1. Materials and solution preparation

Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, Merck, 96%), 3-amino-5-mercapto-1,2,3-triazole (AMTA, Sigma–Aldrich, 96%), absolute ethanol (C<sub>2</sub>H<sub>5</sub>OH, Merck, 99.9%), and acetone (C<sub>3</sub>H<sub>6</sub>O, Merck, 99.0%) were used as received. The high strength steel electrode with a square shape and surface dimensions of 1x1 cm was employed for the electrochemical tests. The chemical composition of the employed high strength steel in this investigation is listed as shown in Table 1. A conventional electrochemical cell accommodates only 200 mL with a three-electrode configuration was used. The three electrodes were

the steel rod, platinum foil, and an Ag/AgCl electrode (in 3.0 M KCl) were used as working, counter, and reference electrodes, respectively. The working electrode for electrochemical measurements were prepared by attaching an insulated copper wire to one face of the sample using an aluminum conducting tape, and cold mounted in resin. The surface of the steel electrode to be exposed to the solution was first ground successively with metallographic emery paper of increasing fineness of up to 600 grits and further with 5, 1, 0.5, and 0.3  $\mu\text{m}$  alumina slurries (Buehler). The electrode was then washed with doubly distilled water, degreased with acetone, washed using doubly distilled water again and finally dried with tissue paper.

**Table 1.** Chemical compositions in wt. % of high strength steel sample that has been used in this study.

Element	C	Si	Mn	P	Cr	Mo	Ni	Cu	Al	Co	B	V	N	Fe
Wt. %	0.03	0.17	0.22	0.01	1.5	0.60	3.2	0.10	0.01	0.01	0.01	0.23	0.02	Rest

## 2.2. Electrochemical measurements

An Autolab Potentiostat (PGSTAT20 computer controlled) operated by the general purpose electrochemical software (GPES) version 4.9 was used to perform the electrochemical experiments. The potentiodynamic polarization curves were obtained by scanning the potential in the forward direction from -1.0 V to -0.2 V vs. Ag/AgCl at a scan rate of 0.001 V/s. The electrochemical impedance spectroscopy (EIS) tests were performed at corrosion potentials over a frequency range of 100 kHz to 100 mHz, with an ac wave of  $\pm 5$  mV peak-to-peak overlaid on a dc bias potential, and the impedance data were collected using Powersine software at a rate of 10 points per decade change in frequency. Each experiment was carried out using fresh steel surface and new portion of the sulfuric acid solution in the absence and the presence of the AMTA molecules. All electrochemical experiments were carried out at room temperature.

## 2.3. Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) investigations.

The SEM images were obtained by using a JEOL model JSM-6610LV (Japanese made) scanning electron microscope with an energy dispersive X-ray analyzer attached for acquiring the EDX analysis.

# 3. RESULTS AND DISCUSSION

## 3.1. Potentiodynamic polarization measurements

The potentiodynamic polarization curves obtained for the high strength steel after its immersion for (1) 30 min, (2) 90 min, and (3) 180 min immersion in 2.0 M  $\text{H}_2\text{SO}_4$  solutions at room

temperature are shown in Fig. 1. These experiments were carried out in order to study the effect of exposure period time on the corrosion behavior of the steel in the sulfuric acid pickling solutions. The values of cathodic ( $\beta_c$ ) and anodic ( $\beta_a$ ) Tafel slopes, corrosion current densities ( $j_{\text{Corr}}$ ), corrosion potential ( $E_{\text{Corr}}$ ), polarization resistance ( $R_p$ ), and corrosion rate ( $R_{\text{Corr}}$ ) obtained from the polarization curves shown in Fig. 1 are listed in Table 2. The values of ( $\beta_c$ ), ( $\beta_a$ ),  $j_{\text{Corr}}$ ,  $E_{\text{Corr}}$ , and  $R_p$  were determined according to our previous study [21,22]. It is well known that the anodic reaction is the dissolution of iron of the alloy, which consumes the electrons produced at the cathode as follows [22]:



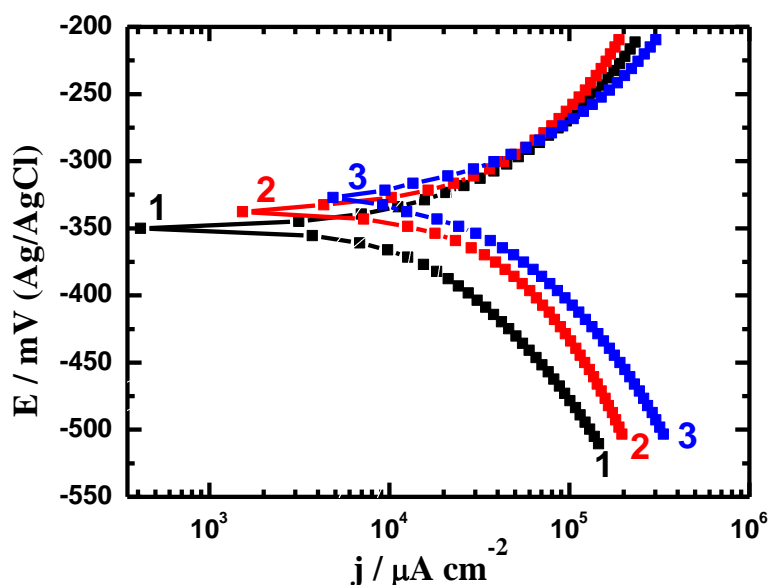
While, the cathodic reaction of steel that consumes the electrons produced by anodic reactions in acid solutions is the hydrogen evolution reaction according to the following equation [22]:



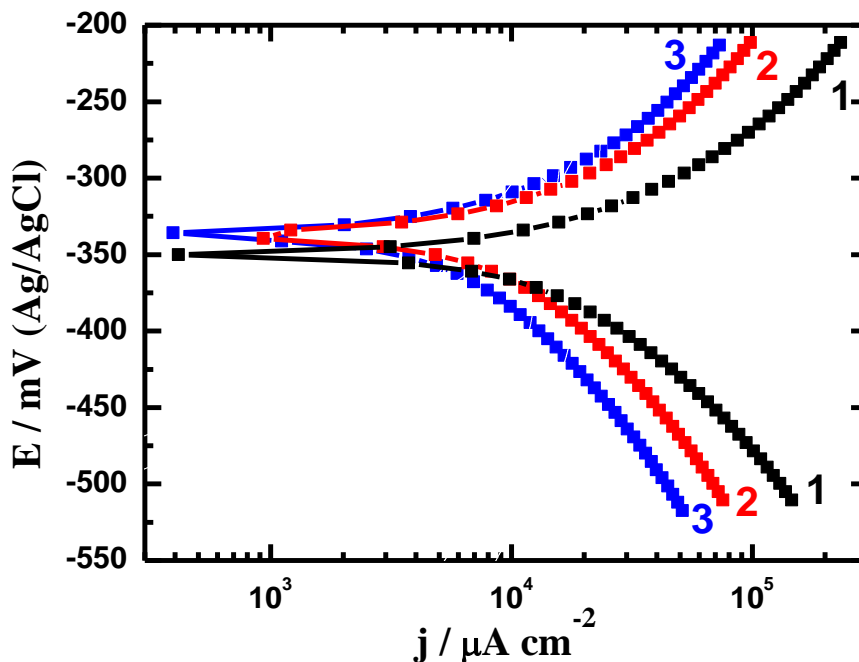
It is obvious from Fig. 1 and Table 2 that increasing the immersion time from 30 min to 90 min and further to 180 min shifts the  $E_{\text{Corr}}$  toward the less negative values, increases the corrosion of the steel through increasing the values of  $j_{\text{Corr}}$  and  $R_{\text{Corr}}$  and decreasing the corresponding  $R_p$  values. This is because increasing the immersion time increases the dissolution of the steel via the corrosion of iron under the continuous attack of sulfuric acid to the alloys surface, which does not allow the alloy to form any oxide and/or corrosion products that can reduce the corrosion [22]. Another reason is that the ferrous cations seen in reaction (1) are not stable and oxidize to ferric cations ( $\text{Fe}^{3+}$ ) as follows [1]:



The severity of these reactions increases with increasing the immersion time, which could lead to the increased dissolution of steel and also explain the increased currents and corrosion rate with increasing the time of immersion. Increasing the applied potential in the less negative direction further facilitates the corrosion of steel as indicated by the increase of the cathodic and anodic currents with potential and with the increase of immersion time.



**Figure 1.** Potentiodynamic polarization curves obtained for the high strength steel after its immersion for (1) 30 min, (2) 90 min, and (3) 180 min immersion in 2.0 M  $\text{H}_2\text{SO}_4$  solutions at room temperature.

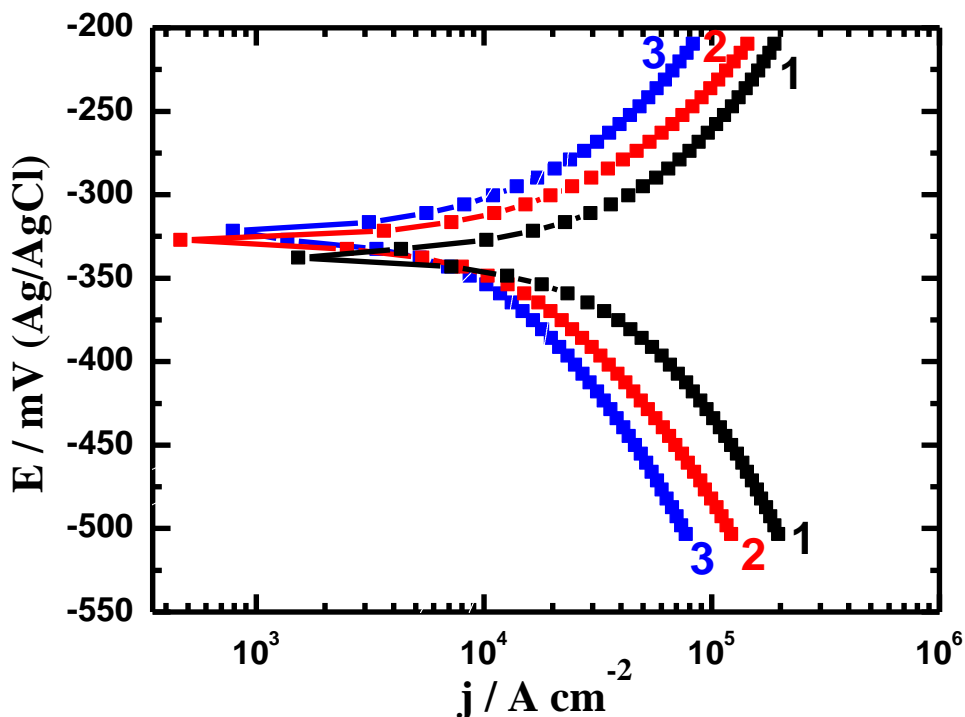


**Figure 2.** Potentiodynamic polarization curves obtained for the high strength steel after its immersion for 30 min in 2.0 M H<sub>2</sub>SO<sub>4</sub> in the absence (1) and the presence of (2) 1x10<sup>-3</sup> M AMTA and (3) 5x10<sup>-3</sup> M AMTA, respectively.

In order to investigate the effect of AMTA on the inhibition of the high strength steel corrosion in the acid solution, the potentiodynamic polarization measurements were also carried out. Fig. 2 shows the polarization curves obtained for the high strength steel after its immersion for 30 min in 2.0 M H<sub>2</sub>SO<sub>4</sub> without (1) and with (2) 1x10<sup>-3</sup> M AMTA and (3) 5x10<sup>-3</sup> M AMTA present, respectively. Similar data were obtained for the steel after 90 min and 180 min immersion in the sulfuric acid pickling solutions in the absence and presence of AMTA molecules and the curves are shown in Fig. 3 and Fig. 4, respectively. The data obtained from Fig. 2, Fig. 3 and Fig. 4 are also listed in Table 2; this is in addition to the values of the inhibition efficiency (IE%) of AMTA as a corrosion inhibitor of the steel in the acidic pickling solution, 2.0 M H<sub>2</sub>SO<sub>4</sub>. The IE% values were obtained according to the following relation [6];

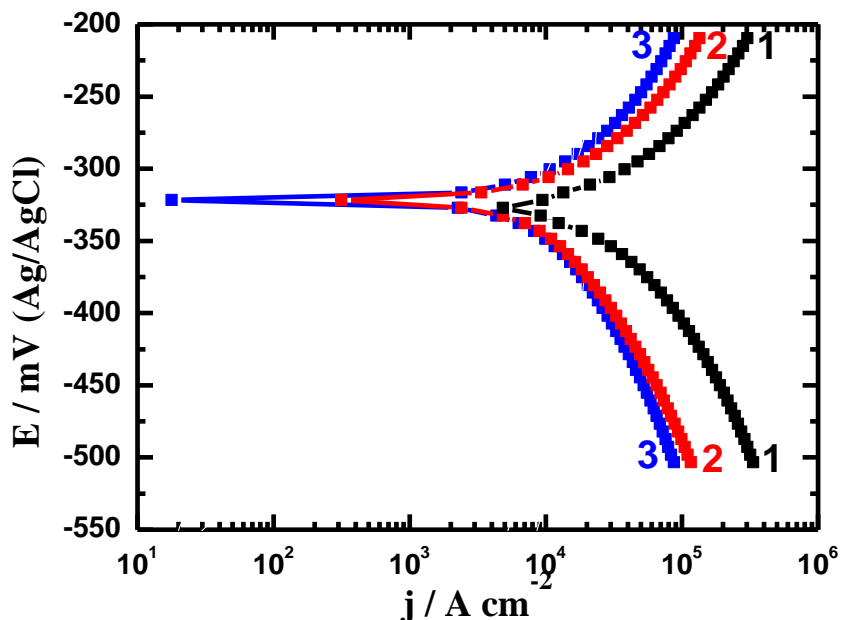
$$IE \% = \frac{R_{Corr}^0 - R_{Corr}^{in}}{R_{Corr}^0} \tag{4}$$

Where  $R_{Corr}^0$  and  $R_{Corr}^{in}$  are the corrosion rates for the high strength steel in the sulfuric acid solution in the absence and presence of AMTA molecules, respectively. It is clearly seen from Fig. 2 that the addition of 1x10<sup>-3</sup> M AMTA within the acid solution (curve 2) after 30 min immersion significantly decreased the cathodic current, anodic current, and corrosion current. This effect was found to highly increase with increasing the concentration of AMTA to 5x10<sup>-3</sup> M (curve 3).



**Figure 3.** Potentiodynamic polarization curves obtained for steel after its immersion for 90 min in 2.0 M  $\text{H}_2\text{SO}_4$  in the absence (1) and the presence of (2)  $1 \times 10^{-3}$  M AMTA and (3)  $5 \times 10^{-3}$  M AMTA, respectively.

Although increasing the immersion time to 90 min and further to 180 min increases the corrosion of the high strength steel, the presence of AMTA and with the increase of its concentration were found to be more effective in inhibiting the steel surface with increasing the time of immersion. This was indicated by the curves obtained after 90 min (Fig. 3) and 180 min (Fig. 4), where AMTA molecules could reduce the anodic current, cathodic current, and  $j_{\text{Corr}}$ . This was further confirmed by the values of  $j_{\text{Corr}}$ ,  $R_p$ , and  $R_{\text{Corr}}$  listed in Table 2, which indicated that the value of  $j_{\text{Corr}}$  and  $R_{\text{Corr}}$  significantly decreased, while the value of  $R_p$  increased upon the addition of AMTA and with the increase of its concentration. Table 2 also shows that the values of the inhibition efficiency recorded for AMTA after longer immersion times were higher than those obtained after 30 min immersion. This indicates that AMTA is a good corrosion inhibitor of the high strength steel in 2.0 M  $\text{H}_2\text{SO}_4$  solution at room temperature and its effectiveness increases with the increase of its concentration to  $5 \times 10^{-3}$  M. This effect for AMTA molecules most probably have resulted due to its adsorption onto the steel surface forming a protective layer that in turn not only isolates the surface but also blocks its active sites and thus precludes the corrosion of the steel in the acid test solution.



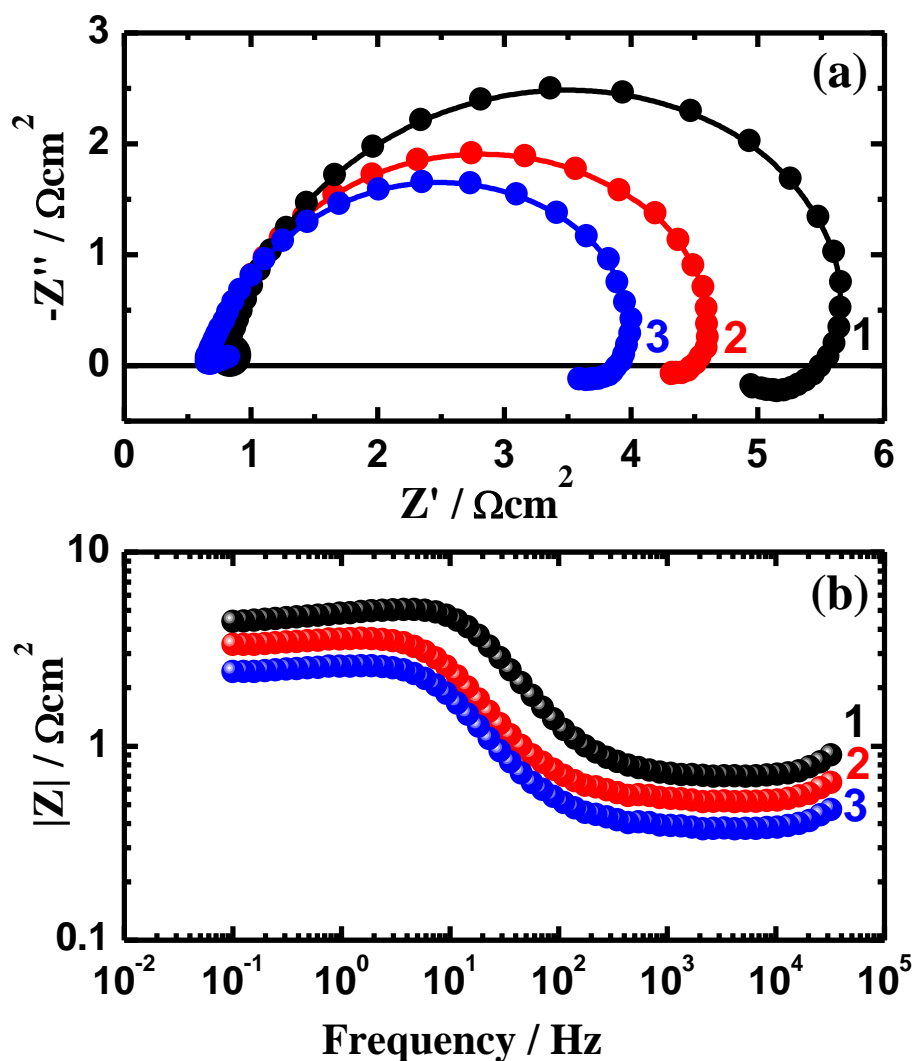
**Figure 4.** Potentiodynamic polarization curves obtained for steel after its immersion for 180 min in 2.0 M H<sub>2</sub>SO<sub>4</sub> in the absence (1) and the presence of (2) 1x10<sup>-3</sup> M AMTA and (3) 5x10<sup>-3</sup> M AMTA, respectively.

**Table 2.** Parameters obtained from the potentiodynamic polarization measurements for the high strength steel electrode that was immersed for different periods of time in 2.0 M H<sub>2</sub>SO<sub>4</sub> solutions in the absence and presence of AMTA molecules.

Medium	Parameter						
	$\beta_c$ mVdec <sup>-1</sup>	$E_{Corr}$ mV	$j_{Corr}$ $\mu A\ cm^{-2}$	$\beta_a$ mVdec <sup>-1</sup>	$R_p$ k $\Omega\ cm^2$	$R_{Corr}$ mmy <sup>-1</sup>	IE %
2.0 M H <sub>2</sub> SO <sub>4</sub> (30 min)	100	-360	13000	95	1.629	15.12	—
+ 1x10 <sup>-3</sup> M AMTA (30 min)	145	-345	8800	100	2.924	10.23	32.53
+ 5x10 <sup>-3</sup> M AMTA (30 min)	155	-343	5500	105	4.948	6.395	57.71
2.0 M H <sub>2</sub> SO <sub>4</sub> (90 min)	105	-338	21000	90	1.003	24.42	—
+ 1x10 <sup>-3</sup> M AMTA (90 min)	130	-335	10800	85	2.069	12.56	48.57
+ 5x10 <sup>-3</sup> M AMTA (90 min)	140	-323	6000	78	3.630	6.976	71.43
2.0 M H <sub>2</sub> SO <sub>4</sub> (180 min)	110	-325	27000	85	0.772	31.39	—
+ 1x10 <sup>-3</sup> M AMTA (180 min)	107	-323	7600	75	2.523	8.836	71.85
+ 5x10 <sup>-3</sup> M AMTA (180 min)	105	-320	6300	70	2.899	7.325	76.67

### 3.2. Electrochemical impedance spectroscopy (EIS) measurements

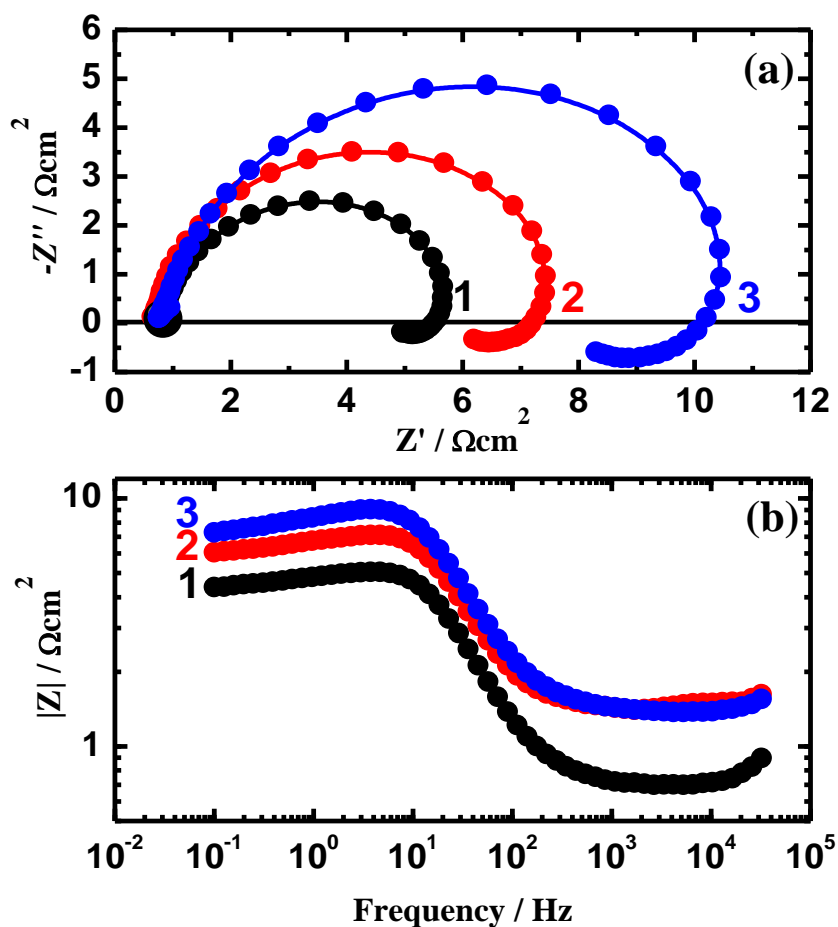
EIS technique has been successfully employed to understand the corrosion and corrosion inhibition for different metals and alloys in corrosive media [6-8,18-20]. Our EIS experiments were carried out to report the kinetic parameters for the steel/ solution interface in absence and presence of AMTA molecules after different immersion periods of time. Fig. 5 shows (a) Nyquist and (b) Bode impedance plots obtained for the high strength steel after its immersion for (1) 30 min, (2) 90 min, and (3) 180 min immersion in 2.0 M H<sub>2</sub>SO<sub>4</sub> solutions, respectively. It is clearly seen from Fig. 5 (a) that the steel in the acid solution shows only one distorted semicircle that its diameter got smaller with the increase of immersion time from 30 min to 90 min and further to 180 min. Fig. 5(b) also shows that the increase of immersion time decreases the impedance of the interface  $|Z|$ , particularly at the low frequency range. This indicates that the increase of the time of immersion increases the dissolution of steel in sulfuric acid solution through decreasing its corrosion resistance [1,24].



**Figure 5.** (a) Nyquist and (b) Bode impedance plots obtained for the high strength steel after its immersion for (1) 30 min, (2) 90 min, and (3) 180 min immersion in 2.0 M H<sub>2</sub>SO<sub>4</sub> solutions.

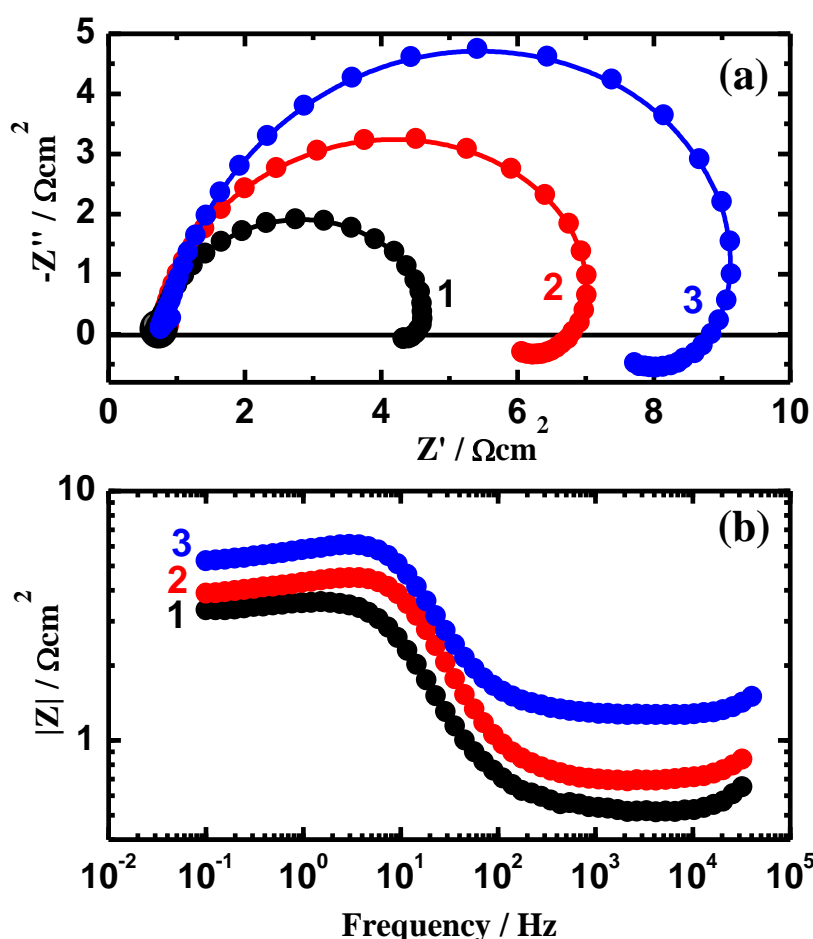


In order to evaluate the effect of AMTA on the inhibition of steel after the different exposure periods of time in the sulfuric acid solution, we also performed the EIS measurements. Typical Nyquist (a) and Bode impedance (b) plots for the high strength steel after 30 min immersion in 2.0 M H<sub>2</sub>SO<sub>4</sub> (1) without and with (2) 1x10<sup>-3</sup> M AMTA and (3) 5x10<sup>-3</sup> M AMTA present respectively are shown in Fig. 6. Similar plots were also obtained for the steel in the acid solution in the absence and presence of AMTA after 90 min and 180 min as shown in Fig. 7 and Fig. 8, respectively. The EIS data were fitted to the best equivalent circuit model that is shown in Fig. 9. This equivalent circuit model was also used to fit the EIS data obtained from studying the corrosion and corrosion inhibition of high strength low alloy steel [1] and maraging steel [6,23] in sulfuric acid solutions. The symbols shown on the circuit model can be defined according to usual convention as following: R<sub>S</sub> represents the solution resistance, Q is the constant phase elements (CPEs), R<sub>p1</sub> is the polarization resistance for the solution/steel interface and can be defined as the charge transfer resistance [25], R<sub>p2</sub> is the polarization resistance for the corrosion product/steel interface, and L is the inductance. The values of these parameters and that were obtained from the fitting process are listed in Table 3.



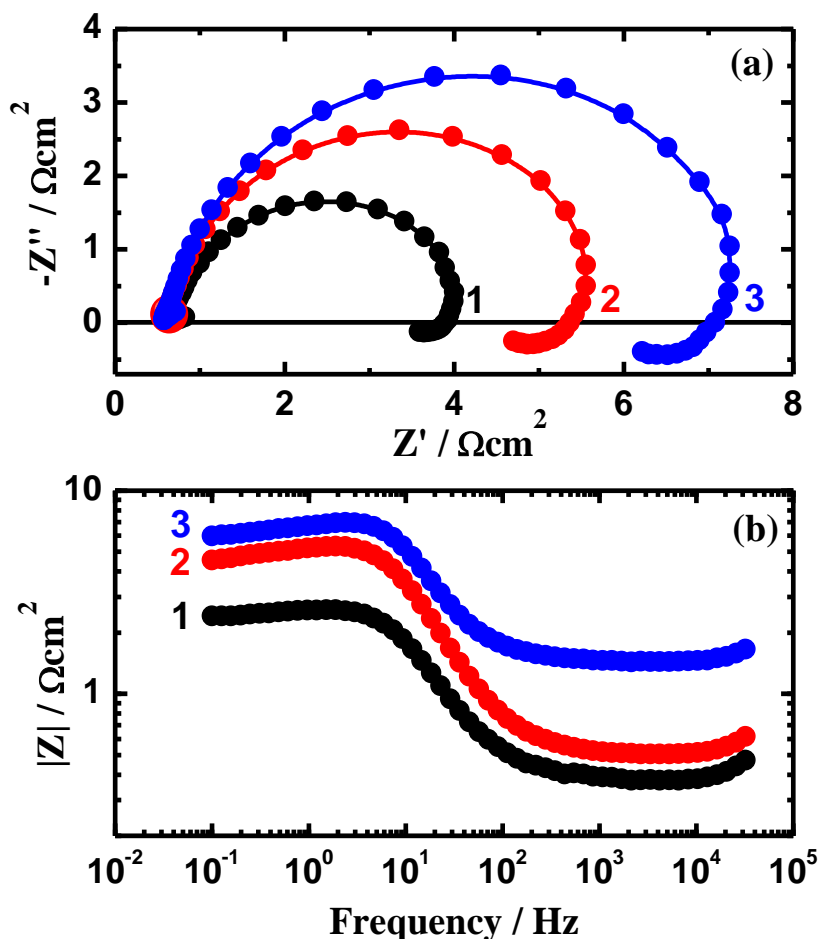
**Figure 6.** (a) Nyquist and (b) Bode impedance plots obtained for the high strength steel after its immersion 30 min in (1) 2.0 M H<sub>2</sub>SO<sub>4</sub> alone and in the presence of (2) 1x10<sup>-3</sup> M AMTA and (3) 5x10<sup>-3</sup> M AMTA, respectively.

It is clearly seen from Fig. 6(a) that the steel in the sulfuric acid alone, curve 1, shows one semicircle that its diameter increases in the presence of  $1 \times 10^{-3}$  M AMTA, curve 2, and further with the increase of its concentration to  $5 \times 10^{-3}$  M AMTA, curve 3. This effect also increased the impedance of the interface as seen from Fig. 6(b). The increase of the diameter of the shown semicircle as well as the impedance of the interface is due to the inhibition of steel corrosion by the presence of AMTA and with the increase of its content in the solution. The data recorded in Table 3 depicted also that the values of  $R_s$ ,  $R_{p1}$ ,  $R_{p2}$  and  $L$  increase, while the value of  $Q$  (CPEs) decrease in the presence of AMTA and with the increase of its concentration. The increase of  $R_s$ ,  $R_{p1}$ , and  $R_{p2}$  indicates that the increased concentration of inhibitor molecules increases the solution and corrosion resistance for the steel surface. The constant phase elements (CPEs,  $Q$ ) with their  $n$  values just less than unity represent double layer capacitors with some pores; the CPEs decrease upon addition of AMTA and upon increase in its concentration, which was expected to cover the charged surfaces to reduce the capacitive effects.

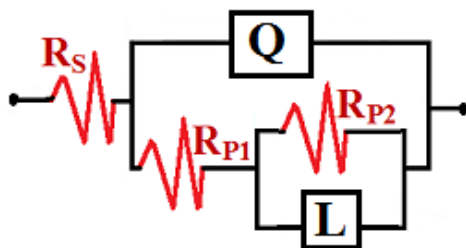


**Figure 7.** (a) Nyquist and (b) Bode impedance plots obtained for the high strength steel after its immersion 90 min in (1) 2.0 M  $H_2SO_4$  alone and in the presence of (2)  $1 \times 10^{-3}$  M AMTA and (3)  $5 \times 10^{-3}$  M AMTA, respectively.

Increasing the immersion time to 90 min (Fig. 7) and further to 180 min (Fig. 8) decreases the diameter of the semicircle and the impedance of the interface whether AMTA is present or not. This confirms also that the increase of immersion time increases the corrosion parameters of the high strength steel in the sulfuric acid solution alone. This was further indicated by decreasing the values of the  $R_s$ ,  $R_{p1}$ ,  $R_{p2}$  and  $L$  and increasing the values of  $Y_Q$  with increasing the immersion time as can be seen from Table 3. On the other hand, the presence of AMTA and the increase of its concentration increased the resistance of the high strength steel against corrosion in 2.0 M  $H_2SO_4$  solutions through increasing the diameter of the obtained semicircle and the impedance of the interface and also by increasing the values of the resistances,  $R_s$ ,  $R_{p1}$ , and  $R_{p2}$ . It is worth to mention also that the efficiency of AMTA as a corrosion inhibitor was found to increase with increasing the immersion time. This was indicated by increasing the values of  $IE\%$  with exposure time as shown in Table 3. The EIS data thus confirm the ones obtained by polarization measurements that the corrosion of the high strength steel increases with increasing time and the presence of AMTA and the increase of its concentration highly inhibit its corrosion in the sulfuric acid pickling solution, 2.0 M  $H_2SO_4$ , particularly with increasing the time of immersion.



**Figure 8.** (a) Nyquist and (b) Bode impedance plots obtained for the high strength steel after its immersion 180 min in (1) 2.0 M  $H_2SO_4$  alone and in the presence of (2)  $1 \times 10^{-3}$  M AMTA and (3)  $5 \times 10^{-3}$  M AMTA, respectively.



**Figure 9.** The equivalent circuit model used to fit the EIS data obtained for the high strength steel after its immersion for different periods of time in 2.0 M sulfuric acid solution in the absence and presence of AMTA molecules.

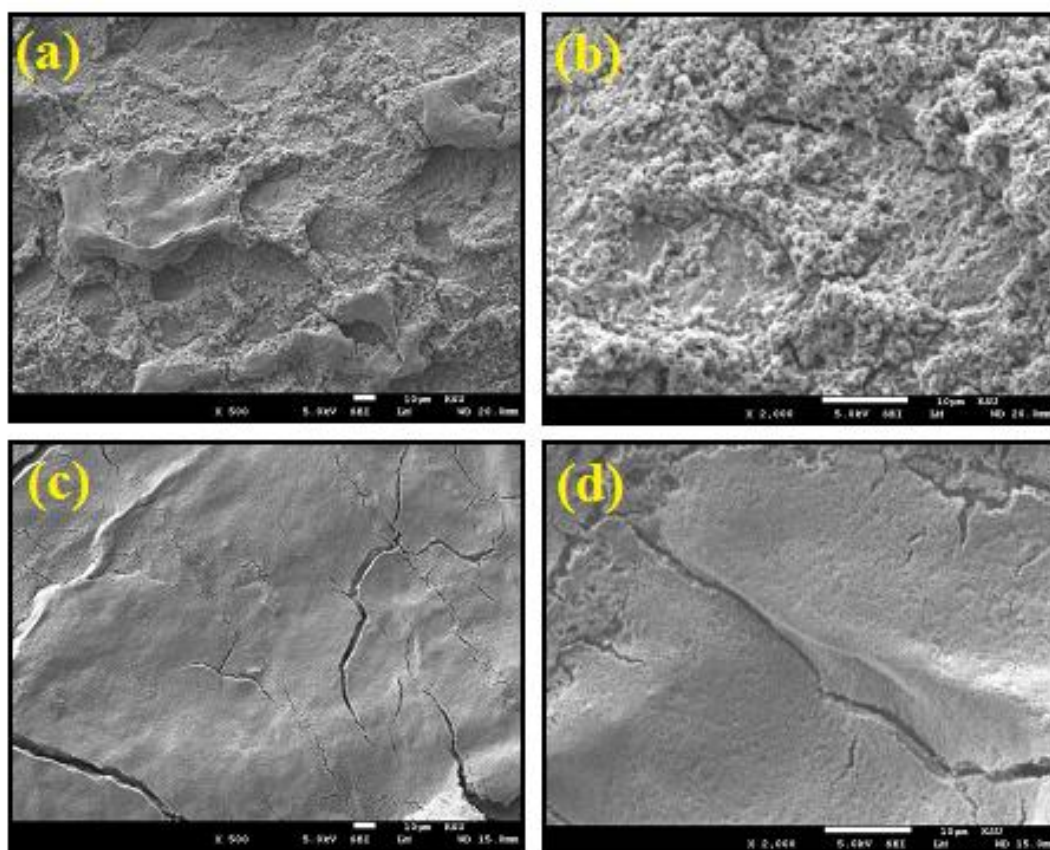
**Table 3.** Parameters obtained by fitting the EIS data with the equivalent circuit shown in Fig. 9 for the high strength steel in 2.0 M  $\text{H}_2\text{SO}_4$  solutions without and with AMTA molecules present.

Solution	Parameter						
	$R_S / \Omega \text{ cm}^2$	$\frac{Q}{Y_Q / \text{F cm}^{-2}}$	$n$	$R_{P1} / \Omega \text{ cm}^2$	$R_{P2} / \Omega \text{ cm}^2$	L / H	IE %
2.0 M $\text{H}_2\text{SO}_4$ (30 min)	1.23	0.00181	0.83	5.988	3.323	15.53	—
+ $1 \times 10^{-3}$ M AMTA (30 min)	1.37	0.00153	0.86	9.653	3.531	15.74	37.97
+ $5 \times 10^{-3}$ M AMTA (30 min)	1.65	0.00138	0.90	13.231	3.745	16.56	54.74
2.0 M $\text{H}_2\text{SO}_4$ (90 min)	1.14	0.00211	0.86	4.876	2.851	14.22	—
+ $1 \times 10^{-3}$ M AMTA (90 min)	1.34	0.00185	0.88	8.741	3.474	15.65	44.22
+ $5 \times 10^{-3}$ M AMTA (90 min)	1.52	0.00163	0.93	11.82	3.850	16.48	58.75
2.0 M $\text{H}_2\text{SO}_4$ (180 min)	1.10	0.00233	0.87	4.294	2.649	13.98	—
+ $1 \times 10^{-3}$ M AMTA (180 min)	1.26	0.00190	0.90	8.482	3.361	15.49	49.38
+ $5 \times 10^{-3}$ M AMTA (180 min)	1.47	0.00174	0.94	11.59	3.747	16.37	62.95

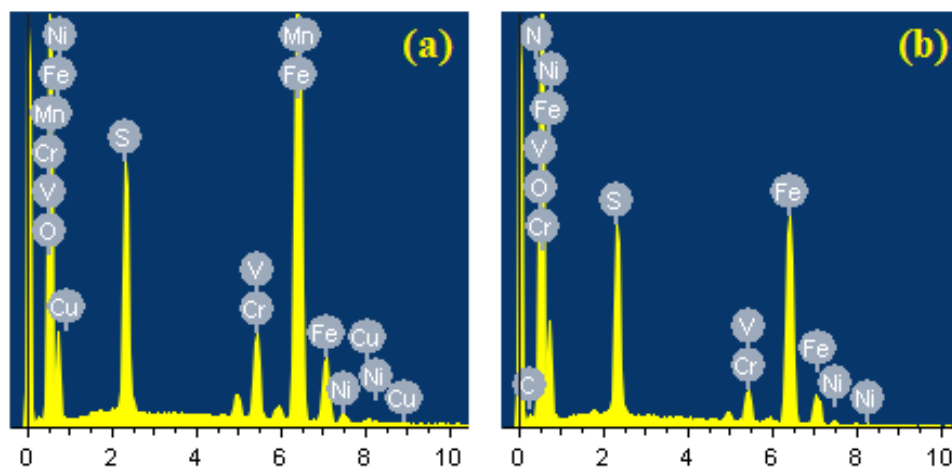
### 3.3. Scanning electron microscopy (EIS) and energy dispersive X-ray (EDX) investigations

In order to shed more light on the effect of  $\text{H}_2\text{SO}_4$  in the absence and presence of AMTA molecules on the corrosion of the high strength steel, SEM micrograph and EDX profile analysis were performed at room temperature. Fig. 10 shows the SEM images obtained for the surface of the high strength steel after 180 min immersion; (a), (b) in 2.0 M  $\text{H}_2\text{SO}_4$  solution alone and (c), (d) in the presence of AMTA molecules, respectively. The SEM micrographs for the steel in acid solution alone, Fig. 10 (a, b) show a total deterioration for the surface, which was due to the aggressiveness attack of the sulfuric acid solution [1,6,23]. On the other hand, the SEM micrograph shown in Fig. 10(c, d) depicted that the surface of the steel is covered with a thick and homogeneous layer of the adsorbed AMTA molecules.

The conclusion driven from the SEM micrographs was also confirmed by the EDX profile analyses shown in Fig. 11, which are corresponding to the high strength steel surface after its immersion for 180 min in (a) 2.0 M  $\text{H}_2\text{SO}_4$  solution alone and (b) 2.0 M  $\text{H}_2\text{SO}_4 + 5 \times 10^{-3}$  M AMTA, respectively. The EDX profile analysis taken for the steel in the acid alone (Fig. 11a) indicated that its surface has the main alloying elements, in addition to the presence of sulfur and oxygen. The presence of O and S was due to the effect of sulfuric acid solutions as well as exposing the steel surface to air after removing it from the acid solution. On the other hand, the atomic percentages of the elements found on the steel surface immersed in 2.0 M  $\text{H}_2\text{SO}_4 + 5 \times 10^{-3}$  M AMTA were 52.88% O, 7.12% S, 0.85% N, 0.76% V, 2.61% Cr, 1.04% Ni, and 28.71% Fe. The presence of nitrogen in the analysis confirms that the AMTA molecules are included in the layer present on the surface. The presence of higher amounts of Cr than that originally present in the steel, in addition to the mighty percents of oxygen indicates that the surface was also passivated through the formation of chromium oxide layer along with the adsorbed layer of the AMTA molecules [1,6,23]. Furthermore, the detection of lower percent of Fe and minute level of Ni revealed that the formed AMTA layer was thick and homogeneously distributed on the surface. This confirms the fact that the inhibition of the high strength steel at this condition is achieved via the adsorption of its molecules onto the metal surface precluding the corrosion of the steel in the high concentrated and aggressive  $\text{H}_2\text{SO}_4$  solution.



**Figure 10.** SEM images obtained for the surface of the high strength steel after 180 min immersion in 2.0 M  $\text{H}_2\text{SO}_4$  solution (a), (b) alone and in the presence of AMTA molecules (c), (d), respectively.



**Figure 11.** The EDX profile analyses obtained on the high strength steel after its immersion for 180 min in (a) 2.0 M  $\text{H}_2\text{SO}_4$  solution and (b) 2.0 M  $\text{H}_2\text{SO}_4 + 5 \times 10^{-3}$  M AMTA, respectively.

#### 4. CONCLUSIONS

The corrosion and corrosion inhibition after 30 min, 90 min and 180 min exposure in 2.0 M  $\text{H}_2\text{SO}_4$  pickling solutions by ATMA at room temperature using electrochemical and spectroscopic techniques have been reported. Potentiodynamic polarization measurements indicated that the increase of the exposure time strongly increases the corrosion of steel as a result of the increased attack of the acid molecules towards the steel surface causing its dissolution. This was confirmed by the increase of corrosion current and corrosion rate as well as the decrease of the polarization resistance with elongating the time of exposure. EIS data also showed that the steel surface and polarization resistances decrease with time due to the inability of steel surface to developing any corrosion product layer under the continuous attack of  $\text{H}_2\text{SO}_4$  molecules. While, the addition of AMTA and the increase of its concentration were found to provide good corrosion inhibition and that effect increased with increasing the immersion time. Where, the presence of AMTA molecules decreased the anodic, cathodic, and corrosion currents, and corrosion rate as well as increased the polarization and solution resistance of the steel in the acid solution. Increasing the concentration of AMTA further increases the inhibition of steel surface. SEM micrographs and EDX analyses confirmed that the inhibition of high strength steel in sulfuric acid solutions is achieved via the adsorption of AMTA molecules onto the steel surface precluding its corrosion. All results confirmed that the high strength steel suffers more corrosion with the increase of its exposure time in acid solution alone and that the presence of AMTA and the increase of its concentration decreases corrosion, particularly with the increase of immersion time.

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