# Study of an Ionic Fluid on the Electrochemical Test of A36 Carbon Steel Ingot

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## Abstract

An ionic fluid based on aromatic heterocyclic family constituted by 1,3-diazole groups was investigated. The purpose is to describe their electrochemical characteristics in order to identify the strategy to avoid the A36 carbon steel surface degradation by using electrochemical measurements. We found that the linear polarization resistance reveals an increasing value when the organic unsaturated cyclic ionic fluid was added to the corrosive electrolyte. The polarization curves and Tafel Extrapolation obtained to know the slopes tafel and the inhibitor efficiency from current density ($i_{corr}$) shows a high efficiency inhibition value.

## Keywords

A36-Steel, Ionic Fluid, Electrochemical Degradation, Tafel Extrapolation, Linear Polarization Resistance, Diazole Group, Organic Inhibitor

## 1. Introduction

One of the primary engineering materials related to the key input for various industries is the steel [1] [2] [3]. To describe their properties and behavior of structural material, steel is assumed, as a polycrystalline solid constituted of a numerous grain in several orientations [4] [5]. These crystalline regions describe a more realistic microalloy chemical element with a specific atomic arrangement without thermodynamic equilibrium conditions. These issues are related not merely to the properties of crystals, equally important are great amount of interesting properties: weldability, hardness, yield strength, toughness, fracture resistance

and, electrochemical degradation [6] [7] [8] [9]. It is well known that the properties are related to chemical elements and their composition. Besides, a chemical association has not been done between the alloying chemical element distribution on surface steel for each phase contented and the molecular interaction of organic compounds in their actuations has retardant of electrochemical degradation.

In particular, electrochemical degradation involves the reactants and surface, taking into account the interface between electrode-electrolyte. In addition, it is generally recognized that the electrochemical process describes a degradation of metallic surface that relates to an intermolecular electron transfer in individual redox reactions [10] [11]. In this work it is argued that electrochemical degradation has become somehow intermediate between atomic arrangement and superficial reactivity. In addition, important considerations, which modify the surface constitution of steel and their inhibitor condition, include type of corrosive medium, and the stability of inhibitor film. Consequently, it is better suited to certain physic-chemical characteristics of the molecule such as functional groups, steric factors, aromaticity, and electron density plus their physical properties like high hydrophobic character as well as high solubility in oil [11] [12] [13] [14]. These parameters, together with the lattice arrangement of atoms and the chemical composition of the metallic steel surface, express a variable behavior.

Based on the assumption that the intrinsic differences in the nature and atomic arrays contribute to the interfacial charge of electrochemical reactions, the inherent microstructure compelling motivation for predicting mechanisms and kinetics of superficial degradation [15] [16] [17]. Following this statement, it is recognized that the susceptibility to steel degradation is one of the very basic problems studied from many paths. Some of the most challenging areas are the cause of metallic deterioration, the consequences of the metals degradation, the expensive replacements, the harmful effects on the environment, the damage-risk of health, and maintenance plus mitigation procedures [18] [19] [20] [21] [22]. The characteristic of chemical mitigations is the use of corrosion inhibitors from different types [23] [24] [25]. It has been reported that chemical corrosion inhibitors are considered the first line of defense that significantly reduces the corrosion rate of metallic materials exposed to corrosion environment [26].

Recently, certain ionic fluids have attained increasing interests for the application as corrosion inhibitors. The interaction between chemical ionic liquid and metallic surface produces a modification of the behavior of both materials [27], this effect allows preventing the danger of devastating failure and the necessity to prolong the component life [28]. Over the recent years, numerous researchers have extended the ionic fluid development to several diverse structure and chemical substances as green inhibitors [29]. The ionic fluids in question it has been contemplated both experimentally and analytically, in which derive through scientific research, there is significant concordance encompassed by various scientists that the molecules with heterocyclic organic compounds plus O, N, or S allows to detailed the control mechanism of inhibiting compounds [29]. In addition,

several studies demonstrated that electronic structure; steric factor, electro density, functional groups molecular area, molecular weight and minor changes in composition of electrolytic solution can significantly change the inhibition effectiveness [29] [30] [31] [32] [33].

With all this in mind, in this work we studied the performance of an organic unsaturated cyclic structure, the 1,3 diazole amino compound. Our synthetic heterocyclic compound is a member to the film inhibitor family; it is capable of forming a protective layer on the steel surface [31] [32]. 1,3 diazole amino compound is designed to such taking account the heterocyclic advantages like molecular receptors for cations and their heteroatom with at least one pair of non-bonding valence shell electrons [15] [27]. In addition, is also considered their high degree of structural diversity presumably to involve a good corrosion protection, and the chemistry-structure related with both the hydrophilicity and hydrophobic property [32]. It is involving the chemical characteristics of 1,3 diazole amino inhibitor, which are together with flow conditions, highly related with their performance and corrosion behavior. With the intent of providing the performance at different dosages, in the laboratory, of our organic compound, the linear polarization resistance (LPR) was used. For this purpose, the ionic fluid selected as inhibitor to mitigate the corrosion was analyzed and compared with results to the analytical studies primarily developed by Mengfei Fan [19]. Finally, the Tafel slope tells us the amount of overpotential needed to increase the reaction rate by a factor ten, and most importantly, allow the corrosion current densities ($i_{corr}$) calculated from the polarization behavior. A brief description of $i_{corr}$ is given when the 1,3 diazole amino is included to electrolyte and compared with $i_{corr}$ results without the organic compound. The analysis is ideally suited to the study of 1,3 diazole amino efficiency and its behavior as an organic chemical inhibitor. We envisage that our results in the corrosion surface inhibition for A36 steel ingot can contribute an appreciated evidence for surface degradation phenomena.

## 2. Details of Experimentation

### 2.1. Materials

A chemical surface protection based on azole organic compound will be studied to enhance the degradation resistance of A36 steel ingot. In keeping with this, a Mexican steel company fabricated the steel, which was prepared by one-pot route with 100% direct reduced iron oxide at 1150°C ± 25°C. The iron oxide in the form of pellets is charged into an electric arc furnace and injected with carbon to react with FeO into the slag and producing CO. After that, the primary melter was poured into a ladle furnace for the secondary steel refining process at a temperature of 1650°C ± 30°C. Secondary metallurgy achieves the removal and dissolution of inclusions, together with the dismissal of gases from liquid steel. Previous to the continuous casting a vacuum degassing was carrying out, in order to remove hydrogen and nitrogen and thus, avoid cracking defects in the

cast. The chemical composition of microalloyed element expressed in wt% of the ingot used is shown in Figure 1, the difference is iron element in wt%.

## 2.2. Preparation of Working Electrode

Preform with working electrode geometry the diameter was 0.5 cm with 1 cm length. The samples were abraded by silicon carbide abrasive papers in a series of grades up to 600 until 1200 #, after that the specimens were polished up to 1 μm diamond finish.

These steel samples used as working electrode were degreased with a mixture of 50:50 acetone and ethanol. Then washed in distilled water and cleaned with deionized water temperature of 25˚C in an ultrasonic bath with a working frequency of 45 kHz during 10 mins. After that, the side and back steel rod surfaces were embedded in epoxy resin, so that the surface area exposed to the electrolyte was limited to 0.5 cm$^2$. An electrical contact was generated using a conductive copper wire that was welded to the back surface enable the electrical conduction of the steel specimens during the electrochemical measurements.

## 2.3. Electrochemical Testing Cell

For electrochemical study, the A36 steel-working electrode was mounted in a specially designed three-electrode cell system allows studying the evaluation of steel specimens by Linear Polarization Resistance method at ambient temperature. In addition, the three-electrode cell allows the potential between the working and reference electrode to be controlled. The cell was made in polycarbonate with an upper inlet and exits through the lower outlet both of 10 mm in diameter; which allow a continuous and fresh electrolyte flow in order to study electrochemical degradation. The electrochemical cell configuration includes a saturated calomel reference electrode (SCE) arrangement for the measurement of corrosion potential ($E_{corr}$), considered standard electrode and characterized as non-polarizable, thus its potential does not fluctuate with the current flow. Thin inert platinum wire with 1 mm diameter and 7 cm length auxiliary electrode was used as counter electrode. This electrode is used to complete the circuit, without no current limitations appear. In addition, allow electrons to flow and delivers a means of applying an input potential to the working electrode.

## 2.4. Preparation of the Reactive Electrolyte

The electrolytic test solution provides a means of ionic composition capable of conducting an electric current. In this research, two types of electrolytes were used. One of them added with the organic unsaturated cyclic structure compounds acting as an inhibitor. The electrolyte test was aerated solution simulated seawater. This mixture was prepared with solute sodium chloride analytical grade reagent purchased from Sigma-Aldrich and distilled water as dissolvent with a primary solution pH of 8.3 consistent with ASTM D1141-98 (2013) [34].
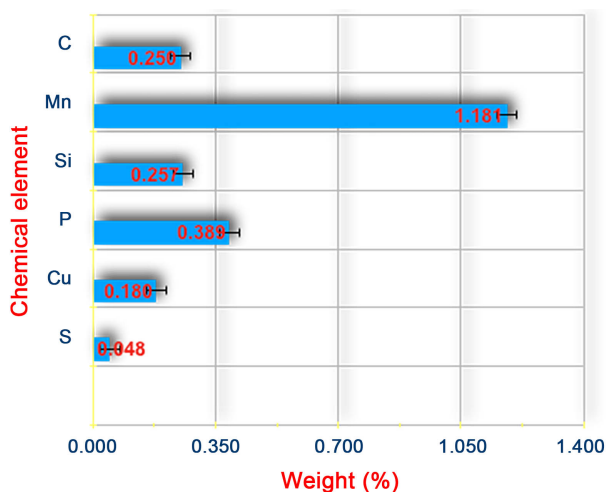
**Figure 1.** Chemical composition of A36 ingot steel in wt% determined by coupled plasma-atomic emission spectroscopy.

The tested electrolyte solution (TES) was also added with a mixture of diesel at 10% with aerated substitute ocean water, besides heated at 60°C plus mechanical agitation for a high-energy emulsification. The temperature was controlled through a heated-circulating bath. After that, the solution was deaerated with analytical grade nitrogen gas bubbling for 120-minutes to remove any oxygen from the electrolyte.

### 2.5. Linear Polarization Resistance Measurements

For purposes of characterizing steel specimen and heterocyclic compound dose the Linear polarization resistance (LPR) was carried out by scanning the current-potential (i-E) domain. Electrochemical tests were performed using a Solartron 1280B electrochemical workstation controlled by CoreView-CoreWare Lab software in a three-electrode cell system. Studies were conducted by anodically polarizing the working steel electrode within the range of −10.0 mV to 10.0 mV vs. $Hg/Hg_2Cl_2$ (SCE) at a scanning sweep rate of 1 mVs$^{-1}$. Furthermore, in order to start with a real work-data, working electrode was immersed in the ionic electrolyte for up to 24 h to demonstrate the time dependence of non-degradation process. In addition, the experimental test was carried out with a low speed stirring of 100 rpm during the saturation of carbon dioxide ($CO_2$), to ensure laminar flow conditions. Three replicate tests of each measurement were performed to ensure reproducibility. Additionally, to establish the importance of the differences among of collecting data, a repeat two-tailed test was conducted. A probability (p) value of 0.05 was reflected to be statistically appropriate.

### 3. Results and Discussion

It is well known that electrochemical degradation occurs when the metallic surface is surrounded aggressive environment rich in ions like anions and cations [22]. In the case of research on ferrous metallic materials, exclusive protection of

natural passivation process throughout protective oxide films is not an option. Therefore, having significant progress the development of chemical compounds with corrosion protection latent like ionic fluids. These chemical compounds are constituted of organic molecules; their contact with ferrous steel surface provides a marked physicochemical reaction [25] [35]. This physic interaction allows inhibiting the degradation surface exposed to corrosive environments in order to prolong the component life. With all this in mind, we determine the polarization resistance ($R_p$) from the slop of the polarization curve obtained from LPR method at the potential interval of ±20 mV relative to $E_{corr}$ vs. open circuit corrosion potential (OCP) and according to the standard ASTMG59. Additionally, LPR is an important source to know the corrosion current density ($i_{corr}$) with regard that galvanic corrosion is an electrochemical process with anodic and cathodic reactions distributed at the corroding surface [36].

$$R_p = \left[ \frac{\Delta E}{(\Delta i)_{\Delta E \to 0}} \right] = \frac{B}{i_{corr}} \qquad (1)$$

where $R_p$ (polarization resistance) is the ratio between the potential shift $\Delta E$ and the corresponding current change $\Delta i$ in an experimental polarization,

$$B = \left[ \frac{(\beta_a)(\beta_c)}{2.303(\beta_a + \beta_c)} \right] \qquad (2)$$

While $B$ is a constant determined by the slopes $\beta_a$ and $\beta_c$ of the Tafel lines of the anodic and cathodic partial reactions. The resistance obtained represents the equilibrium conditions, where the electrochemical reaction only depends on the exchange current density.

Figure 2, current densities of A36 steel are given in two different environmental conditions based on two types of electrolytes. Into one of the aqueous electrolytes the heterocyclic diazole compound was dissolved. Which means this ionic fluid solution could decrease the dissolution rate of the A36 working electrode. The results show a disturbing of the electrochemical degradation process, where the ionic fluid act as inhibitor. The concentrations selected, could affect the transfer of electrons between steel surface and electrolyte environment with the consequent slow kinetics of the corrosive reactions. The corrosive electrolytic test solution was dosed with the heterocyclic compound at a concentration of 20 ppm. Physical mixture of amino ionic fluid compounds was prepared with a pure 1-propanol as dissolvent at 50°C ± 2°C. Prospects of actually prevent electrochemical degradation of steels by chemical corrosion inhibitors considered the first line of defense. Ionic fluid solutions could effect on reduces the corrosion rate of metallic materials exposed to corrosion environment.

Focused on the electrochemical properties of our working electrode based on interface steel and electrolyte, the linear polarization resistance reveals a concordance with previous investigations [37] [38]. Thus, despite significant progress on organic unsaturated cyclic structure behavior as corrosion inhibitor, numerous challenges still exist for reveling the internal phase microstructure to act
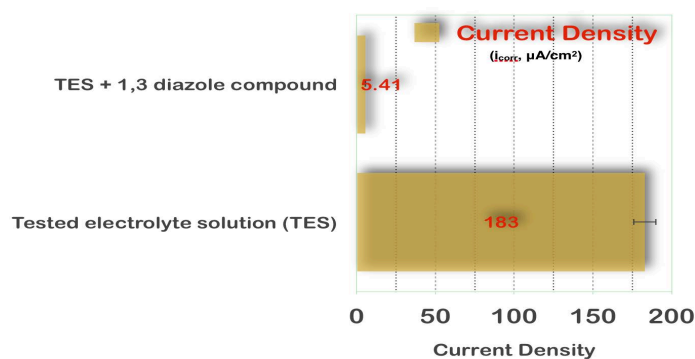
**Figure 2.** Effect of electrolyte on the current densities ($i_{corr}$, $\mu$A/cm$^2$) measurements, which are based on environmental parameters like the presence of 1, 3 amino diazole.

synergistically with chemical components and their relationship with the molecular interaction of the organic unsaturated cyclic compound. With this in mind, **Figure 3** refers that $R_p$ tend to be more sensitive to high immersion time, with significant effects on the electrolyte with the organic unsaturated cyclic imidazole added. Although the published studies lack data physical details of the concentration used to understand the chemical reactions on the steel surface and organic compound with inhibition effect [39]. In this research, the polarization resistance ($R_p$) to surface oxidation when the electrolytic has been mixed with organic unsaturated cyclic imidazole shows an increasing resistance of 11.63, 19.84, and 22.55 times higher. These data compared with just artificial seawater electrolyte both at the same duration 1, 12 and 24 h of immersion time, respectively. It should be noted that the ionic fluid or organic imidazole compound provides a protective ability to prevent the electrochemical degradation of the A36 steel surface. To investigate the variation in the $R_p$ it was considered the time parameter to analyze the results obtained. This study has revealed that the highest $R_p$ value was obtained after 24 hours of immersion time. Such value suggests a possible correlation within the formation a more compact inhibitor layer [40] on the A36 steel surface. Two types of surface phenomena physisorption or chemisorption could form a protective layer [41] [42]. Even if here we are addressing the general aspect of adsorption, the most relevant benefit of organic compound layer relief onto promotes a resistance to degradation or oxidation process. Several reactions are convincingly based on the electron transfer, mainly at the A36 steel surface; of particular interest are iron ions originated during their interaction in presence of environment, which can be corrosive or just react with electrolyte, taking account a steel surface reduced with the consequent metallic dissolution. This kind of reaction is related to corrosion parameters like reaction velocities, the essential data of electrode-electrolyte phenomena have been associated with the rate of dynamic oxidization and $R_p$, which quantitatively explain the inverse dependence between the surface degradation with the resistance to surface oxidation [43].
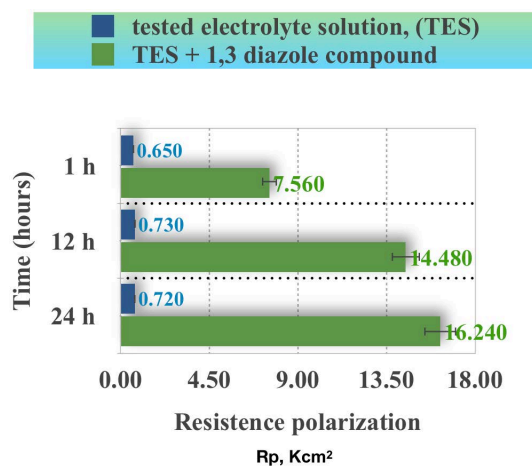
**Figure 3.** The influence of corrosive environment on the surface of A36 steel. During its acting as anode under the electrochemical degradation process.

Understanding the chemical reactions between ions and electron interaction when significant electrolyte mixtures involved provide an important consideration against corrosion. With this in mind, in **Figure 4** should be noted the results of polarization resistance monitoring the possible corrosion resistance of A36 steel under study in an artificial sea aqueous environment and also with the enriched aqueous environment when the ionic fluid organic compound is added.

It is interesting to mention that the polarization curves describe a significant diminution of the electrochemical oxide and reduction reactions because the organic diazole compound was mixed with the artificial sea aqueous electrolyte. It should be noted that comparisons with the polarization resistance curve of measurements just with artificial sea electrolyte revealed a dynamic electrochemical degradation performance. Note, these results could be associated with an increase of current density for the anodic region indicating electrochemical dissolution of the A36 steel and evidently a poor corrosion resistance. It is obviously clear that when heterocyclic organic compound are present in the artificial-sea aqueous electrolyte, it acts as a barrier that interferes with the steel surface and aggressive environment deceleration the electrochemical corrosion experienced by the A36 steel. In order to remarks our results, it should be considered a previous work, in which the electrochemical degradation include the absorption and localized diffusion of chemical anions on the steel surface. The chemical ions with their effective negative charge influence the electrochemical activity in the aqueous phase and its tendency toward defective sites on steel surface [44].

This relationship, it is feasible to understand when a comparative surface energy among crystalline solid phases is present like in our A36 steel-working electrode. In the face of these facts, the overall reaction indicates an electrochemical redox process, which imposes consistency of corrosion degradation in the absence of the organic compound [45]. In order to evaluate the consequence
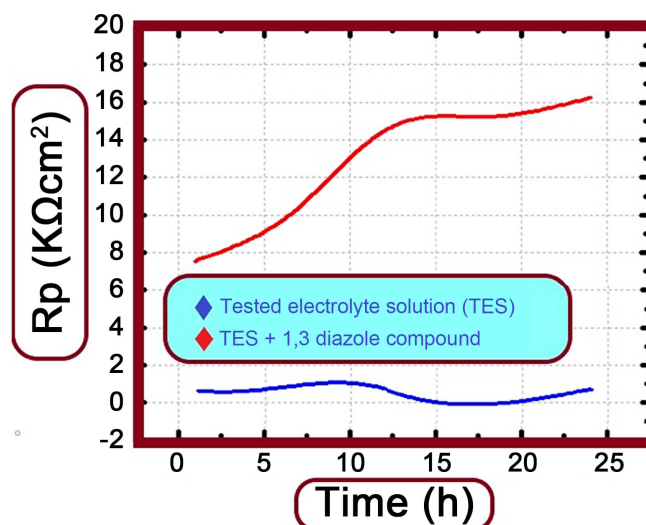
**Figure 4.** Corrosion resistances for A36 steel working electrode under study and the relation with the environment.

of organic compound added to the electrolyte solution was calculated the percentage inhibition efficiency from polarization measurements using the following equation.

$$\eta_p = \left(1 - \frac{i}{i_o}\right) \times 100\%$$

where $i$ and $i_o$ are the corrosion current density values in the presence and absence of organic compound respectively.

The efficiency value was 97.04%, which shows the performance of organic compound avoiding the electrochemical degradation of metallic A36 steel surface in contact with the electrolyte. Furthermore, the $\eta_p$ suggest an elevated inhibition behavior influenced by the characteristics of the organic molecule, although there are so many aspects and chemical elements answerable for oxidative degradation [46].

**Figure 5** shows the structural representation of the imidazole organic compound, some delicate balance concerning with their functional group, steric factors, aromaticity, and electron density, just to name a few, play an important role in the inhibition performance [47]. Some results have concluded that the functional groups have a specify interaction with the metal surface [23] [26].

It is well known that, properties of substance depend on the chemical structure, in our case, the organic compound posses a heterocyclic five-membered rings, in which one of the two nitrogen heteroatoms has one pair of non-bonding valence shell electrons, besides the amine group is strong electron donor. It is expected that the –C=N– group contribute decreasing the electrochemical degradation.

Due to the electronegative nitrogen in the molecule could be a strong interaction with the A36 steel surface. At this point, it is possible that imidazole layer exposure of the steel by adsorption of the organic compound molecules [48].
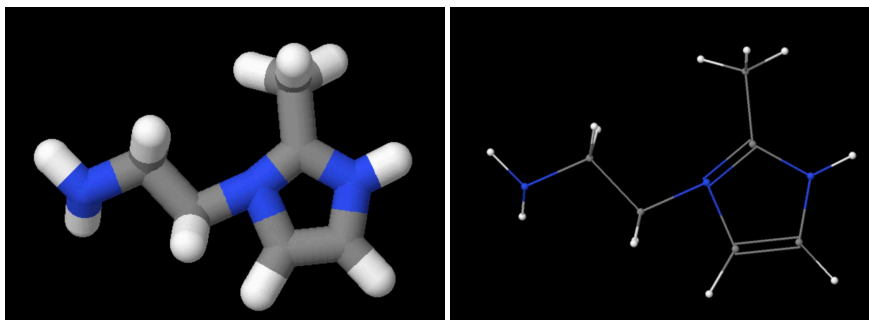
**Figure 5.** Chemical structural representation of [1-(2-aminoethyl)-2-metil]-1,3-diazacyclopenta-2,4-diene. Besides, the functional group NH2 involves the availability of an aromatic pi-cloud [15].

While generous work has been realized to identify the surface tension and thermodynamic [49] [50] properties of the ionic fluid organic compound the degradation rate has been also influenced by the chemical composition of environment like acidity or salinity among others.

However, exist for reveling the energy of internal microstructure including the alloy chemical components of A36 steel to act synergistically with the environment and the ionic fluid organic compound against the electrochemical degradation of the steel surface. Therefore, it is fundamental and profitable, taking account the polycrystalline microstructure integrated by diverse orientation grains.

These crystalline grains describe a specific atomic arrangement that should provide the chemical influence of electrochemical degradation [22]. Furthermore, according to the fluctuations on chemical content in each internal region from phase, precipitations, and grain boundary to general microstructure should be a particular influence interaction of the heterocyclic organic molecules to obstruct the electrochemical degradation.

Obtaining quantitative information of current density when the imidazole molecules are present on the corrosive electrolytic led to the development of decreased value (**Figure 6**).

A reasonable explanation for the $i_{corr}$ decrement suggests that organic compounds interact with electrolyte molecules, which promote less surface degradation [51]. It is found that the dosage or organic compound tends to decrease the iron dissolution present as working electrode [52]. Evidently it suggests that this kind of organic compound with nitrogen heteroatom can be strongly adsorbed on the steel surface, and reduce the corrosion attack.

Additionally, some important results reported that ionic fluids are highly followed for good inhibition efficiency, particularly the fluid ionics containing nitrogen, sulfur, and oxygen like alkylimidazolium ionic liquids [53]. To seek a deeper understanding of the heteroatom influenced some investigations revealed that several bonds comprehend their adsorption relies on covering the metallic surface [54] [55].
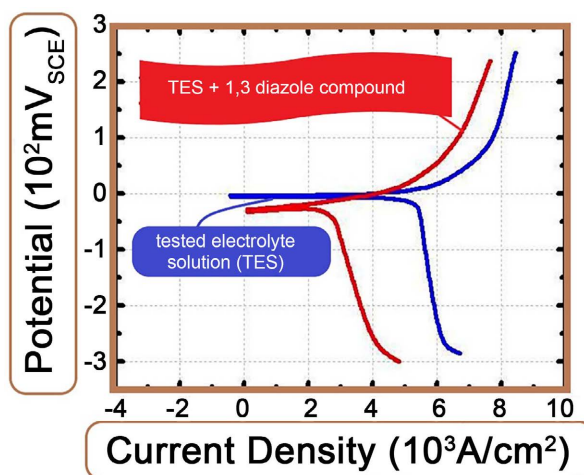
**Figure 6.** Polarization curves for A36 steel immersed in electrolytic solution in the presence and absence of inhibitor solution.

## 4. Conclusions

The framework of heterocyclic organic compound added to corrosive environment encompasses with the behavior of electrochemical polarization tested just with the electrolyte solution. With detailed tests, we show that the ionic fluid with organic compound added in 20 ppm describes an increased polarization resistance. Relating with an effective suppress of the electrochemical degradation. The fact that we have found a high efficiency value of 97.92%, demonstrates the effectiveness against the electrochemical degradation.

This work reveals that the investigated ionic fluid heterocyclic organic compound added in a concentration below 50 ppm is an appropriate inhibitor of electrochemical degradation protecting the steel surface.

## Acknowledgements

## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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