

Electrochemical analysis of the corrosion resistance of annealed steel with nickel coating in marine environment simulations: chronoamperometric and voltametric study

Análisis electroquímico de la resistencia a la corrosión del acero recocido con recubrimiento de níquel en simulaciones de ambiente marino: estudio cronoamperométrico y voltamétrico

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ABSTRACT

This study conducts a comprehensive investigation into the corrosion resistance of annealed steel wire, commonly used in marine construction, emphasizing its importance for the material's durability and functionality in such applications. An innovative approach was implemented by applying a Nickel (II) Chloride Hexahydrate coating through electrodeposition. This process was meticulously adjusted, varying critical parameters such as exposure time and electrical potential. To mimic marine corrosive conditions, a Sodium Chloride solution was used, allowing for a detailed comparative analysis between coated and uncoated samples. The evaluation was based on rigorous quantitative measurements of the wire's surface state and corrosion potential, using TAFEL curves for precise interpretation. The findings empirically demonstrated that the coating significantly enhances corrosion resistance, markedly outperforming untreated steel. These results reinforce the proposition of nickel electrodeposition as an effective and viable strategy for corrosion protection in marine industrial applications.

Keywords: Corrosion resistance; annealed steel wire; marine construction applications; electrodeposition process.

RESUMEN

Este estudio lleva a cabo una investigación exhaustiva sobre la resistencia a la corrosión del alambre de acero recocido, comúnmente utilizado en la construcción marina, enfatizando su importancia para la durabilidad y funcionalidad del material en dichas aplicaciones. Se implementó un enfoque innovador mediante la aplicación de un recubrimiento de hexahidrato de cloruro de níquel (II) mediante electrodeposición. Este proceso fue ajustado meticulosamente, variando parámetros críticos como el tiempo de exposición y el potencial eléctrico. Para imitar las condiciones corrosivas marinas, se utilizó una solución de cloruro de sodio, lo que permitió realizar un análisis comparativo detallado entre muestras recubiertas y no recubiertas. La evaluación se basó en mediciones cuantitativas rigurosas del estado de la superficie del cable y el potencial de corrosión, utilizando curvas TAFEL para una interpretación precisa. Los hallazgos demostraron empíricamente que el recubrimiento mejora significativamente la resistencia a la corrosión, superando notablemente al acero sin tratar. Estos resultados refuerzan la propuesta de la electrodeposición de níquel como una estrategia eficaz y viable para la protección contra la corrosión en aplicaciones industriales marinas.

Palabras claves: Resistencia a la corrosión; alambre de acero recocido; aplicaciones de construcción marina; proceso de electrodeposición

INTRODUCTION

Metallic material corrosion presents a continual challenge in industrial and technological fields [1]. This phenomenon, involving the degradation of metallic materials through chemical and electrochemical reactions with their environment [2], significantly threatens the durability and performance of metal components and structures across various applications [3]. Exposure to corrosive environments, notably marine conditions, is a particular concern due to its potential to accelerate corrosion and reduce material lifespan [4-5]. This study addresses this issue by focusing on the evaluation and mitigation of corrosion in annealed steel wires. It employs a comprehensive method involving the application of a Nickel (II) Chloride Hexahydrate coating via electrodeposition. Nickel (II) Chloride Hexahydrate has been utilized in electrodeposition, as demonstrated in studies for creating Ni-B composite coatings on steel substrates [6]. This approach promises to enhance the corrosion resistance of steel wires while preserving the structural integrity of steel and other materials [7-10]. Marine corrosion conditions were simulated using a Sodium Chloride solution, mimicking seawater's chemical properties [11]. Advanced electrochemical analysis techniques, such as chronoamperometry and linear voltammetry, were employed to characterize corrosion kinetics [12]. This study aims to develop sophisticated strategies in corrosion protection for annealed steel materials. The hypothesis suggests that nickel electrodeposition is highly effective in preventing corrosion, potentially impacting the durability and resilience of these materials in corrosive environments. Advanced electrochemical techniques, including chronoamperometry and linear voltammetry, provide an in-depth understanding of corrosion kinetics. These insights pave the way for future research and improvements in the efficiency and durability of industrial products.

METHODOLOGY

The assessment of corrosion in annealed steel wires and the subsequent implementation of a Nickel (II) Chloride Hexahydrate coating represents a significant advancement in the field of electrochemistry and corrosion protection. This methodology has been designed considering the latest research and cutting-edge techniques to achieve highly precise and relevant results [13]. The key stages include:

Sample Selection and Preparation:

High purity annealed steel wires were selected with very rigorous specifications, following standardized protocols. The composition and characteristics of the selected SAE 1006 steel wire, which is a low carbon steel known for its malleability and ductility, making it suitable for applications like annealed wire, are detailed in Table 1. The sample preparation included an exhaustive cleaning stage, focusing on the elimination of oxides, surface contaminants, and potential impurities that might affect the coating's adhesion.

Nickel (II) Chloride Hexahydrate Electrodeposition Process:

The electrodeposition process was carried out using a 0.1 Molar solution of Nickel (II) Chloride Hexahydrate (99.9%, Sigma-Aldrich) in Potassium Chloride (99%, Sigma-Aldrich). This represents a highly effective approach in corrosion mitigation, based on previous research highlighting the efficacy of this chemical combination. The concentration and temperature (25°C) were precisely controlled, and the electrochemical assays were performed using an AUTOLAB PGSTAT302N Metrohm® potentiostat/galvanostat, rigorously calibrated and synchronized with a computer system equipped with NOVA software for precise data acquisition and analysis. Emphasis is placed on ensuring consistency and reproducibility of results, with all potential measurements systematically referenced to the standard Ag/AgCl electrode. The electrochemical setup consisted of a Working Electrode (WE) made of annealed steel wire, a Reference Electrode (RE) of Ag/AgCl for maintaining a constant reference, and a Counter Electrode (CE) of vitreous carbon, selected for its stability and reliability in these measurements. This electrode configuration was carefully chosen to optimize the precision and reliability of the assays, critical aspects for the integrity of the collected data and its subsequent scientific interpretation.

Oxygen can act as an unwanted oxidizing agent, affecting both the kinetics of corrosion reactions and the interpretation of electrochemical data. In this context, nitrogenation is presented as an essential technique to establish a controlled, oxygen-free environment. [14-16]. High-purity nitrogen was used to displace dissolved oxygen from the solution in the electrochemical cell. This process was carried out for 5 minutes, ensuring adequate saturation and efficient removal of oxygen. The stability of the inert environment was verified through measurements of dissolved oxygen, ensuring that the level was insignificant for corrosion reactions. By avoiding oxygen interference, more representative data of intrinsic corrosion processes are obtained, allowing a more accurate assessment of the corrosion resistance of materials, as well as the electrochemical behavior of corrosion inhibitors.

Potentiostat Programming for Chronoamperometry: Chrono-amperometry, a fundamental technique in corrosion electro-chemistry, requires meticulous programming of the potenti-ostat to obtain precise and reproducible measurements.

Potentiostat Configuration: The potentiostat is set to apply a constant potential to the working electrode. The accuracy of the applied potential is critical, and feedback control is used to ensure potential stability over time.

Measurement of Temporal Current: The current is monitored and recorded as a function of time. This current profile provides vital information about the dynamics of electron transfer processes and corrosion reactions.

Data Analysis: Chronoamperometry data are analyzed to extract kinetic parameters, such as reaction rate constant and corrosion current density. This information is fundamental to understanding corrosion mechanisms and designing effective corrosion protection strategies.

Table 1. Characteristics of Tramontana® Annealed Steel Wire (ASTM A853 Standard, SAE 1006 Composition)

Diameter (mm)	Tensile Strength (kg/mm ²)	Carbon (%)	Phosphorus (%)	Manganese (%)	Sulfur (%)
1.65	32	.08	.04	0.4	.05

Source: own elaboration

Simulation of Corrosion Conditions in Seawater:

To emulate practical conditions, samples were immersed in a 3.5% solution of Sodium Chloride (99%, Sigma-Aldrich),

simulating the corrosive characteristics of seawater [17-19]. This stage takes into account the influence of environmental variables that are critical for corrosion in real-world applications. The same type of electrodes continued to be used.

Electrochemical Analysis:

In this study, advanced techniques of chronoamperometry and linear voltammetry were implemented, using a high-precision Metrohm® potentiostat/galvanostat. The electrodes used, as described earlier, were fundamental to ensuring the integrity of the obtained data. These methodologies allowed for detailed acquisition of information on the electrochemical kinetics of corrosion and facilitated the quantitative evaluation of key parameters. The notable resolution and sensitivity of the equipment used ensured the reliability and reproducibility of the results.

The importance of this research lies in its focus on the effectiveness of Nickel (II) Chloride Hexahydrate coatings in mitigating corrosion on annealed steel wires. The findings of this study are highly relevant for various industrial applications and represent a significant advancement in corrosion protection in adverse environments.

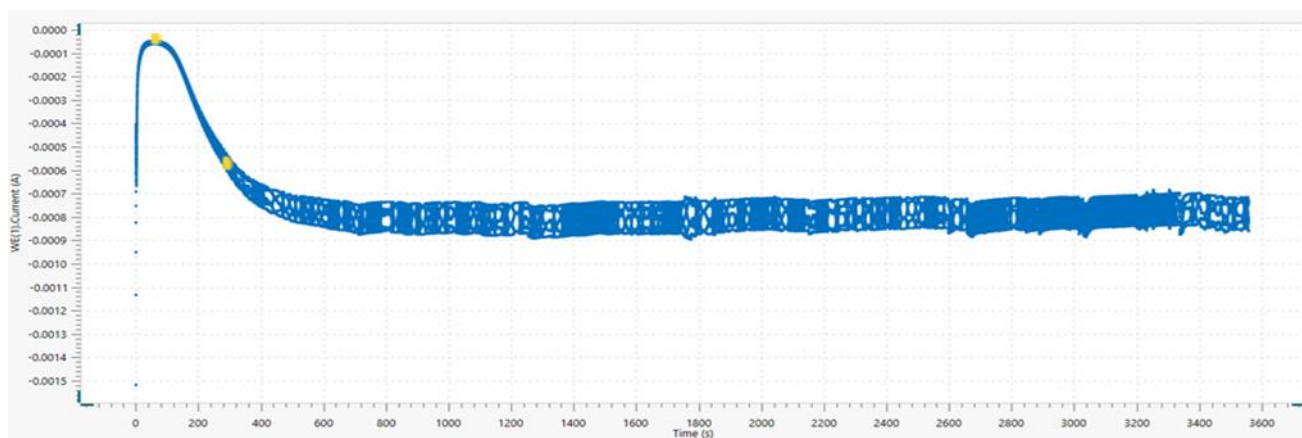
From a methodological standpoint, chronoamperometry was applied for a detailed analysis of the electrodeposition of Nickel (II) Chloride Hexahydrate on annealed steel wire. This electrochemical analysis was performed using a working electrode composed of the annealed steel wire, a platinum chloride reference electrode, and a vitreous carbon counter electrode. This approach allowed for a deep understanding of the kinetics and efficiency of the nickel electrodeposition process. Experiments were conducted over two different time intervals, obtaining electrodeposition samples on the annealed wire at 3600 seconds and another at 300 seconds, in addition to maintaining control samples without undergoing electrodeposition.

Linear voltammetry was subsequently applied to these three categories of samples. The potentiostat was programmed to operate for 300 seconds with a potential oscillating between -1.5 to 1.5 Volts, and a scan range of 0.01 Volts/second. These conditions were replicated in the three samples, providing essential information for the comparative evaluation of the effects of electrodeposition.

RESULTS

The results obtained from this cutting-edge study conclusively indicate the efficacy of Nickel (II) Chloride Hexahydrate coating in mitigating corrosion on annealed steel wires. The investigations were carried out with precision and rigor, and the findings are highly significant in terms of industrial applications and corrosion protection. Key results are presented below. Figure 1 shows a chronoamperometry capturing the behavior of the electric current over time during the electrodeposition of Nickel (II) Chloride Hexahydrate on the annealed steel wire. This technique provides a window into the dynamics of electrodeposition and the formation of a nickel layer on the wire surface. A detailed interpretation follows.

Figure 1. Chronoamperometry of the nickel electrodeposition process on an annealed steel electrode over 3600 seconds



Source: own elaboration

Start of the Current: The process begins with an initial current of approximately -0.0015 amperes. This initial phase is characterized by rapid adhesion of nickel ions to the active areas of the steel wire. The high initial current suggests the presence of numerous available sites for deposition.

Increase in Current (0-40 seconds): During the first 40 seconds, the current experiences a rapid increase, exceeding -

0.0001 amperes. This rise can be attributed to the initial deposition of a nickel layer on the wire's surface. The uniformity of this stage suggests that the annealed steel wire possesses a structure conducive to uniform electrodeposition.

Current Decay (40-300 seconds): In the subsequent period, the current gradually decreases, reaching values near -0.0006 amperes. This decay reflects the formation of a denser and more homogeneous nickel layer on the wire. The current reduction indicates a decrease in available active sites on the surface.

Stabilization and Disturbances (300-3600 seconds): The current stabilizes at around -0.0008 amperes and experiences disturbances, varying between -0.009 and -0.007 amperes. These fluctuations result from ion redistribution processes and the dynamics of the solution around the working electrode.

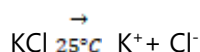
The choice of the platinum chloride reference electrode ensures accurate measurements of the electrochemical potential throughout the process. The use of the vitreous carbon counter electrode, inert in this context, prevents any interference with nickel electrodeposition.

The results obtained from the chronoamperometry of Nickel (II) Chloride Hexahydrate electrodeposition on annealed steel wire shed light on the kinetics and efficiency of the process. The rapid initial adhesion of nickel ions translates into an increase in current, indicating uniform deposition. As time progresses, the formation of a denser nickel layer decreases the electrochemical activity of the surface.

This experimental approach, with an emphasis on appropriate electrode selection, can be applied in future studies to understand and optimize electrodeposition on various materials and substrates.

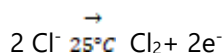
In this Nickel (II) Chloride Hexahydrate electrodeposition system on annealed steel wire, potassium chloride (KCl) plays an essential role. KCl acts as an electrolyte, providing potassium (K^+) and chloride ions (Cl^-), enabling electrical conduction in the solution. This is crucial for the electrodeposition process, as efficient electrical current flow between electrodes is necessary for the desired reaction to occur.

KCl dissolution breaks down into potassium and chloride ions as per the following equation:



Reaction (1)

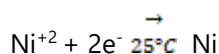
When an electrical potential is applied in the electrochemical cell, chloride ions (Cl^-) move towards the counter electrode (graphite) and undergo an oxidation reaction:



Reaction (2)

This reaction produces chlorine gas (Cl_2) at the counter electrode. Chlorine is a byproduct of electrodeposition and is not directly related to nickel deposition on the working electrode (annealed steel wire). Instead, nickel is reduced to its metallic form and deposited on the working electrode as per the equation:

Nickel Reduction Reaction:

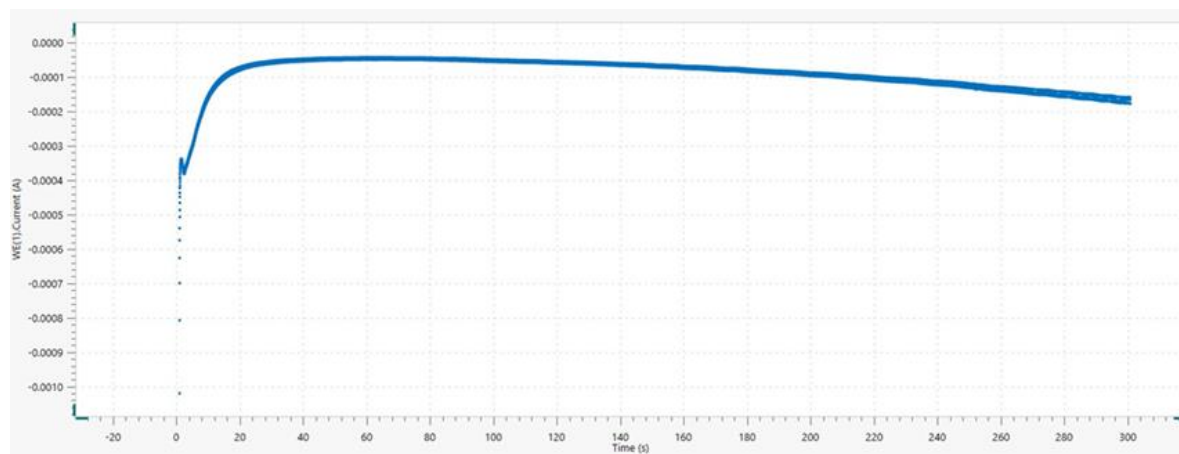


Reaction (3)

This reduction process of nickel ions (Ni^{+2}) allows nickel to adhere to the steel wire during electrodeposition. Although the steel wire is electrically neutral overall, the applied electrical potential creates a localized "polarization" on the wire's surface, facilitating the adhesion of nickel ions. This adhesion is a fundamental part of the electrodeposition process and allows for selective nickel deposition on the steel wire.

In summary, potassium chloride (KCl) in the solution serves as an ionic conducting medium, allowing ions to flow between electrodes during the electrodeposition process. Chloride ions (Cl^-) are oxidized at the counter electrode, generating chlorine gas, while nickel ions (Ni^{+2}) are reduced at the working electrode, depositing metallic nickel on the annealed steel wire surface.

This process is essential for forming the nickel coating on the wire and ultimately for enhancing its corrosion resistance. In the 300-second chronoamperometry shown in Figure 2, the high initial current reflects rapid nickel ion reduction but decreases due to the formation of a dense nickel layer on the electrode surface. This limits ion arrival at the electrode-solution interface.

Figure 2. Chronoamperometry of the Nickel Electrodeposition Process on an Annealed Steel Electrode over 300 Seconds

Source: own elaboration

Meanwhile, in the 3600-second chronoamperometry previously shown in Figure 1, the current stabilizes at -0.0008 amperes, indicating that the reduction rate equals the nickel ion diffusion rate. A uniform nickel layer has limited the reaction rate to the ion transport speed.

Oscillations and Transients: The 300-second chronoamperometry shows initial oscillations, suggesting transient adsorption and desorption of nickel ions on the electrode surface due to the variability of the initial nickel layer. At 3600 seconds, the oscillations have dampened, indicating stability in the nickel layer quality and reaction kinetics.

Observation Time and Stability: The 300-second chronoamperometry captures the early stages of nickel layer formation. The 3600-second chronoamperometry provides an extended view, where the nickel layer becomes uniform and stable. **Scientific Importance:** The 3600-second chronoamperometry offers more precise data and establishes a balance between reaction kinetics and mass diffusion. The 300-second chronoamperometry is valuable for identifying initial behaviors but does not provide information on the steady state of the process.

After successfully completing chronoamperometry tests of 3600 seconds on a variety of materials and analyzing the resultant electrochemical responses, we progressed to an additional stage of study. In this phase, we performed linear voltammetry on both the materials previously subjected to 3600-second chronoamperometry and those that underwent a 300-second chronoamperometry. We also included materials that were not subjected to chronoamperometry, i.e., those without nickel electrodeposition, yielding the following samples:

Samples 1 (3600s Chronoamperometry - Nickel Electrodeposition on Annealed Steel Wire): In this test, a 3600-second chronoamperometry was applied to study the nickel electrodeposition on annealed steel wire, with several runs to obtain consistent data.

Samples 2 (3600s Chronoamperometry - Nickel Electrodeposition on Annealed Steel Wire): Similar to Test 1, a 3600-second chronoamperometry was performed to investigate the nickel electrodeposition on annealed steel wire, with multiple executions.

Samples 3 (Samples without Nickel Electrodeposition): In this test, chronoamperometry was not conducted, resulting in samples without nickel electrodeposition. Multiple executions were carried out to validate the results.

Linear Voltammetry

To simulate sea water conditions, we prepared a solution containing approximately 3.6% NaCl. With utmost care, we introduced this solution into an electrochemical cell, using the same electrodes made of the material of interest that were employed in the previous phases of our study.

The potentiostat was meticulously configured, setting a time of 300 seconds and a potential range varying from -1.5 V to 1.5 V, with a constant scan rate of 0.01 Volts per second. This protocol was applied in the three previously mentioned tests, and the detailed results are presented in Figure 3. Specifically, Figure 3a shows the linear voltammetry corresponding to Test 1, Figure 3b presents the linear voltammetry for Test 2, and Figure 3c exhibits the voltammetry applied to the materials without nickel coating, i.e., in Test 3.

As shown in Table 2, the combination of chronoamperometry and linear voltammetry provides a more

comprehensive understanding of how nickel electrodeposition affects the surface area and properties of annealed steel materials in the various samples under simulated marine corrosion conditions.

Samples 1 (3600s Chronoamperometry - Nickel Electrodeposition on Annealed Steel Wire): This series of tests focused on studying the nickel electrodeposition on annealed steel wire through the application of 3600-second chronoamperometry in multiple executions. This extensive duration of chronoamperometry allowed for a detailed analysis of the nickel electrodeposition process on the annealed steel material. Subsequently, linear voltammetry was performed on these samples in a 3% sodium chloride solution, designed to simulate corrosion conditions in marine environments. The results demonstrated a 6% increase in surface area compared to its initial state.

Samples 2 (300s Chronoamperometry - Nickel Electrodeposition on Annealed Steel Wire): Similar to Test Series 1, this series focused on the nickel electrodeposition on annealed steel wire, but with the application of 300-second chronoamperometry on multiple occasions.

This variation in the duration of chronoamperometry allowed for a comparison of results with Test Series 1 and an evaluation of how different electrodeposition times affect the process and outcomes. Subsequently, linear voltammetry was performed on these samples in a 3% sodium chloride solution, designed to simulate corrosion conditions in marine environments, and it was observed that the surface area remained consistent with its initial state.

Samples 3 (Samples without Nickel Electrodeposition): In this series of tests, chronoamperometry was not conducted, resulting in samples that did not undergo the nickel electrodeposition process. Multiple executions were carried out to ensure the consistency of results and to validate the importance of electrodeposition in the behavior and properties of annealed steel materials. Subsequently, linear voltammetry was applied to these samples in a 3% sodium chloride solution, designed to simulate corrosion conditions in marine environments, revealing a 27% decrease in surface diameter compared to its initial state.

Table 2. Results of Linear Voltammetry and Changes in Surface Area in Sample

Tests	Nickel Electrodeposition Performed Previously	Initial Diameter in cm	Final Diameter after Linear Voltammetry in cm	Difference in cm	Percentage (%)
Sample 1 (With 3600 s Chronoamperometry -- Nickel Electrodeposition on Annealed Steel Wire)	Si	0.5566	0.5908	-0.0342	6
Sample 2 (360 s Chronoamperometry - Nickel Electrodeposition on Annealed Steel Wire)	Si	0.5566	0.5566	0	0
Sample 3 (Samples without Nickel Electrodeposition)	No	0.5566	0.4056	0.151	-27

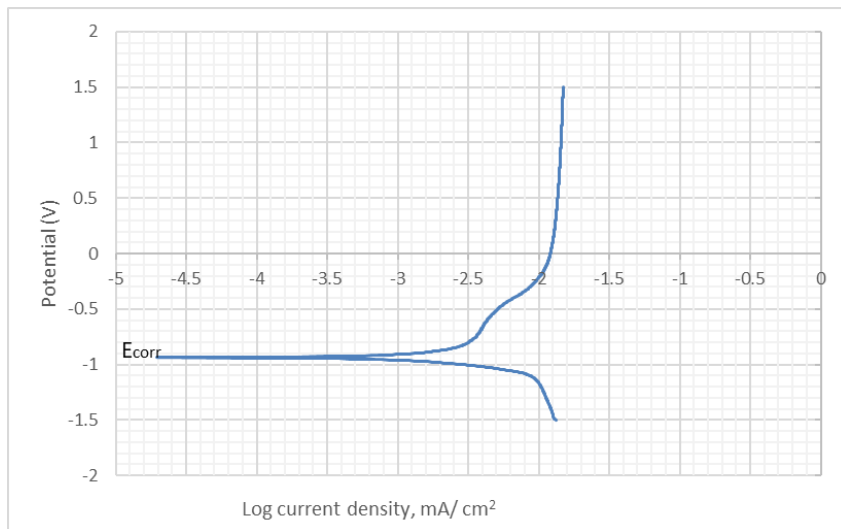
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Transformation of Linear Voltammetry Results into Tafel Plots: The generation of Tafel plots from Linear Voltammetry results is a fundamental process in modern electrochemical research. This advanced technique is grounded in principles established in high-impact scientific literature, enabling the extraction of detailed information about the electrochemical behavior of materials in simulated corrosive environments.

In analyzing linear voltammetry data, normalization methods and calculations based on current density are applied, which facilitates the generation of Tafel curves. These curves provide critical information about corrosion potential (E_{corr}) and corrosion rate in material systems subjected to corrosion in aggressive environments, as illustrated in figures 3 (a-c)

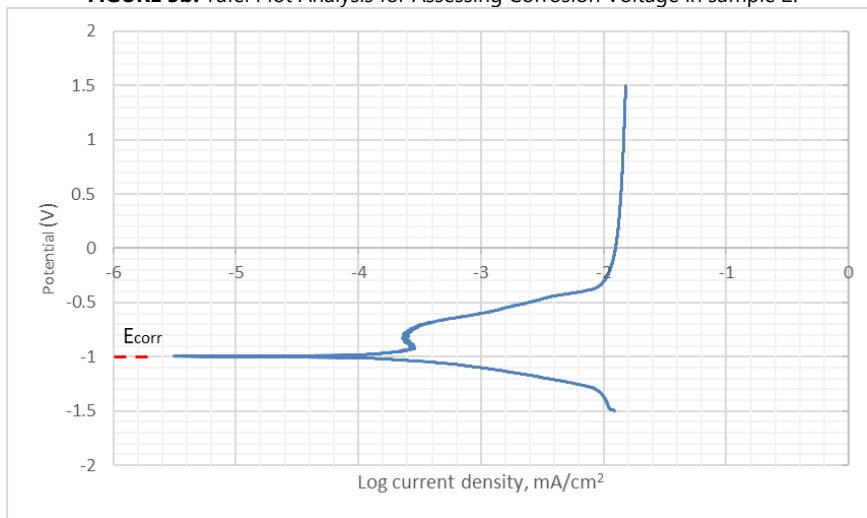
The Tafel plot technique has been extensively validated in renowned electrochemical studies, and its application in this study enhances the understanding of the electrochemical response of annealed steel wires, both with and without nickel coating, in simulated marine conditions. These results hold significant relevance for both industry and materials research, contributing to the advancement of knowledge in the field of corrosion protection.

FIGURE 3a. Tafel Plot Analysis for Assessing Corrosion Voltage in sample 1



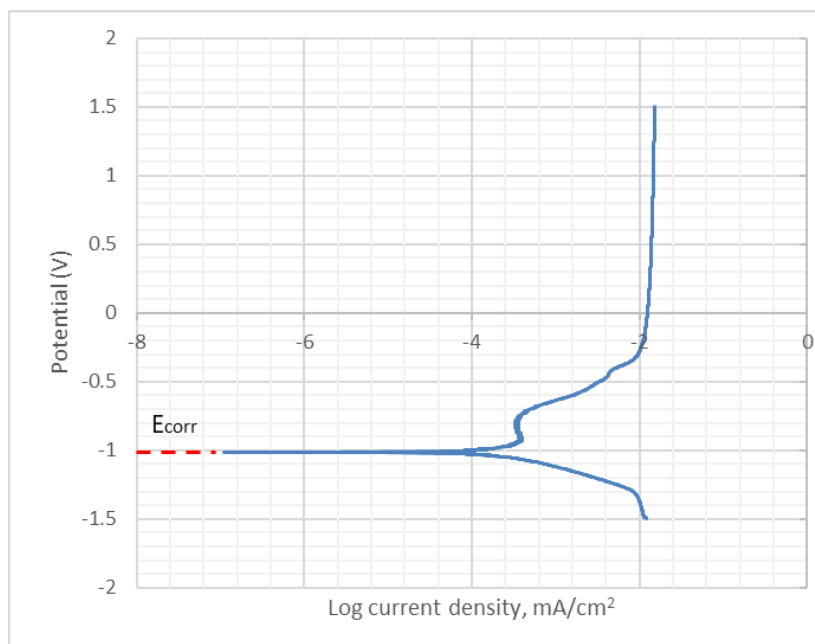
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FIGURE 3b. Tafel Plot Analysis for Assessing Corrosion Voltage in sample 2.



Source: own elaboration

FIGURE 3c. Tafel plot analysis for assessing corrosion voltage in sample 3.



Source: own elaboration

As shown in Table 2, the experimental setup involved configuring the potentiostat for a duration of 300 seconds and a potential range spanning from -1.5 to 1.5 V. The potential sweep rate was consistently maintained at 0.01 Volts per second. For the analysis of results, it is necessary to consider that, to evaluate the electrode properties in relation to the kinetic parameters of the electrochemical reactions in which these electrodes are involved, it is beneficial to examine their behavior with respect to Tafel plots; that is, establishing a linear association between the potential applied to the electrode/electrolyte interface and the logarithm of the current density referred to the actual unit area of the electrode.

A more negative voltage in the oxidation region indicates a faster oxidation rate, implying that metal corrosion is occurring at a high rate at that potential.

From the results of the linear voltammetry figures, it is deduced that the most negative corrosion potential belongs to the sample without nickel electrodeposition, while Sample 2 is less negative at -0.99700928 voltage, being the one with an intermediate range of nickel deposits and subjected to a chronoamperometry range of 300 seconds, for the case where electrodeposition of 3600 seconds was performed (S/R, 300s). Meanwhile, the other two samples also exhibit negative numbers, but these are below -1, as shown in Table 2.

CONCLUSIONS

The nickel electrodeposition on annealed steel wires has proven to be a highly effective strategy in corrosion prevention. The findings of this study corroborate that this coating process can significantly enhance the corrosion resistance of metallic materials.

Increase in Surface Area: Beyond its capability to thwart corrosion, it has been observed that nickel electrodeposition leads to an increase in surface area in certain cases. This augmentation may have positive implications in terms of the durability and mechanical strength of the treated materials.

Electrochemical Discernment: The Tafel plots derived from linear voltammetry results have provided insightful information about the corrosion potentials in each type of steel wire. This knowledge is pivotal for understanding and managing corrosive processes in engineering systems.

Industrial Relevance: The insights gained from this study bear substantial industrial significance, suggesting that nickel electrodeposition can considerably extend the lifespan of metallic materials in corrosive environments. This could lead to significant enhancements in the efficiency and longevity of products and structures used across various industrial applications.

Future Prospects: This work lays the groundwork for future research in the field of corrosion protection and metal material coating. Opportunities arise to explore novel coating formulations, variations in electrodeposition processes, and applications across a broad spectrum of industries.

In summary, this study represents a substantial advancement in the understanding of corrosion protection through nickel electrodeposition on annealed steel wires. The results obtained have profound implications in both the academic realm and industry, offering a promising path toward more resilient and enduring materials in corrosive environments.

LIMITATIONS

Marine Conditions Representativeness: A significant limitation is how accurately our laboratory simulations represent the complex real marine conditions. Incorporating additional variability in environmental factors in future studies would be beneficial.

FUTURE ENDEAVORS

Development of Advanced Coatings: Exploring multi-layer or composite coatings that enhance corrosion resistance and other mechanical properties could offer significant advancements.

Fatigue and Fracture Analysis: It is vital to conduct studies on the coating's impact on material fatigue resistance and susceptibility to fracture under cyclic loads.

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