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Abstract of the Ph.D. Thesis

Anti-Corrosive Coatings based on Polymers and Nanomaterials

Ph.D Student

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Keywords: corrosion protection; zinc substrate; silica coatings; graphene oxide; modified graphene oxide; epoxy coatings; silica nanoparticles; corrosion inhibitor; electrochemical impedance spectroscopy (EIS); polarization curves; photocatalytic activity; temporary coating

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1. Introduction

Zinc is the world's fourth most used metal, after iron, aluminum, and copper. Notably, more than 60% of global zinc production is dedicated annually to protecting steel from corrosion [1]. According to the International Lead and Zinc Study Group, approximately half of the annual zinc consumption is used for galvanic protection of steel [2]. This widespread use highlights the importance of zinc in various industries, including automotive and construction, where it is a vital element in protective steel coatings [3]. Galvanized coatings are particularly effective in resisting atmospheric corrosion, although rapid degradation can occur in highly polluted environments. Additionally, zinc shows good corrosion resistance in freshwater; however, its effectiveness diminishes in seawater. Since the introduction of the galvanization process, the corrosion behavior of zinc has been extensively studied under various environmental conditions [4].

2. State of the Art

Corrosion Inhibitors

Corrosion inhibitors can be classified based on their chemical structure into inorganic and organic corrosion inhibitors, or based on their development stage, into traditional and modern corrosion inhibitors [5]. Corrosion inhibitors mitigate metal degradation through an electrochemical mechanism by effectively suppressing the anodic and/or cathodic reactions involved in the corrosion process. This process blocks active sites, modifies anodic and/or cathodic potentials, and may create a protective barrier or film. Depending on their function, these inhibitors can be classified as anodic, cathodic, or mixed-type inhibitors [6]. Corrosion inhibitors are widely used as a corrosion protection strategy, favored due to their ease of use, efficiency, and low cost.

Corrosion-Resistant Coatings

Protective coatings are the most commonly used method for preventing corrosion [7]. They form a protective layer that acts as a barrier, shielding the material from its surrounding environment [8], by preventing water and oxygen from reaching the surface [9]. However, research suggests that the effectiveness of barrier protection depends on the coating's ability to prevent ion penetration [10]. The barrier coating's ionic impermeability maintains a high electrical resistance to moisture at the coating-substrate interface. As a result, the electrolyte solution's conductivity at the substrate remains low, effectively reducing the transfer of corrosion current between the anode and cathode [9]. These coatings can be categorized into four types: metallic, inorganic, organic, and inorganic-organic hybrid coatings. Adequate corrosion protection requires resistance to environmental factors, long-term stability, and strong adhesion to the substrate [7].

4. Thesis Objectives

This thesis aims to develop, characterize, and evaluate advanced corrosion-resistant coatings for zinc substrates, focusing on (i) silica-based coatings, containing functionalized graphene, (ii) epoxy coatings, and (iii) chitosan-based coatings. The research systematically optimizes coating performance by integrating graphene oxide into silica matrices, enhancing epoxy-based coatings with functionalized nanomaterials, developing a temporary coating, and exploring environmentally friendly corrosion inhibitors.

The systems discussed in this thesis can be broadly categorized into two main groups: inorganic coatings on zinc, which feature a silica matrix, and organic coatings on zinc, comprising epoxy and chitosan layers. For each type of coating, some *specific objectives* were surveyed, as outlined below:

A) Electrochemical Evaluation of the Relationship between the Thermal Treatment and the Protective Properties of Thin Silica Coatings on Zinc Substrates

- Formation of silica layers *via* the sol-gel method and the dip-coating technique.
- Emphasis on the critical role of temperature in silica network formation and its impact on coating performance.
- Determining optimal heat treatment parameters for preparing thin silica (SiO_2) coatings on zinc.
- Optimization of drying temperature and duration to enhance corrosion resistance.
- Evaluation of coatings through electrochemical impedance spectroscopy (EIS), potentiodynamic polarization, and morpho-structural characterization.

B) Corrosion Behaviour of Zinc Coated with Composite Silica Layers Incorporating Poly(amidoamine)-modified Graphene Oxide

- Modification of graphene oxide (GO) with poly(amidoamine) (PAMAM) dendrimer to improve dispersion in silica coatings.
- Investigation of the effect of GO-PAMAM incorporated into silica matrices on the corrosion behavior of zinc.
- Comprehensive characterization of graphenes using FT-IR, Raman spectroscopy, TEM, and XRD.
- Comparative analysis of SiO_2-GO-PAMAM coatings against SiO_2 coatings with GO, reduced graphene oxide (rGO), and APTES-modified GO.
- Examination of silica sol-ageing influence on coatings' anti-corrosive properties, emphasizing the role of polycondensation.
- Morphological analysis conducted *via* SEM, with wettability assessed through contact angle measurements.

C) Correlations between the Anti-Corrosion Properties and the Photocatalytic Behavior of Epoxy Coatings Incorporating Modified Graphene Oxide Deposited on Zinc Substrate

- Development of a zinc-epoxy resin substrate-coating system incorporating modified graphene oxide (GO) for improved corrosion resistance and photocatalytic properties.
- Correlation between anti-corrosion performance and photocatalytic behavior of the coatings.
- Application of thin epoxy (EP) layers containing 0.1 *wt*% GO, rGO, APTES-modified GO, or PAMAM-modified GO onto zinc substrates *via* dip-coating.
- Electrochemical impedance spectroscopy (EIS) study.
- Morphological and structural analysis of coatings and modified GO particles.

D) Epoxy Coatings Doped with (3-Aminopropyl)triethoxysilane-Modified Silica Nanoparticles for Anti-Corrosion Protection of Zinc

- Development of epoxy coatings incorporating silica (SiO_2) and (3-Aminopropyl)triethoxysilane-modified silica (SiO_2-APTES) nanoparticles on zinc substrates *via* dip-coating technique.
- Exploration of two nanoparticle embedding methods: (I) incorporation of APTES-modified silica nanoparticles into epoxy resin, and (II) in-situ functionalization of silica nanoparticles in epoxy gel before hardener addition.
- Characterization of nanoparticles using Fourier-Transform Infrared Spectroscopy (FT-IR) and Transmission Electron Microscopy (TEM), and evaluation of coatings via Scanning Electron Microscopy (SEM), Energy-Dispersive X-ray Spectroscopy (EDS), contact angle measurements, and adhesion tests.
- Evaluation of corrosion resistance using electrochemical impedance spectroscopy and polarization curves.

E) Temporary Anti-Corrosive Double Layer on Zinc Substrate Based on Chitosan Hydrogel and Epoxy Resin

- Development of chitosan/epoxy double-layer coatings for zinc substrates for temporary and peelable-on-demand corrosion protection.
- Assessment of coating thickness and adhesion properties.
- Application of Raman spectroscopy for evaluation of organic layer interactions.
- Evaluation of pseudo-porosity in the coatings.
- Application of electrochemical impedance spectroscopy (EIS) for corrosion resistance evaluation and analysis of electrolyte nature influence.

F) New Eco-Friendly Corrosion Inhibitor for Zinc based on Expired Detralex Drug. Adsorption and Electrochemical Studies

- Use of expired Detralex as a green corrosion inhibitor for zinc.
- Electrochemical investigation of Detralex as a corrosion inhibitor for zinc protection using electrochemical impedance spectroscopy, polarization curves, and cyclic voltammetry.

- Application of scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDS) for surface morphology observation of zinc electrodes.

- Adsorption Behavior of Detralex on Zinc Surface and Isotherm Model Evaluation.

5. Electrochemical Evaluation of the Relationship between the Thermal Treatment and the Protective Properties of Thin Silica Coatings on Zinc Substrates [11]

In this study, we conducted a detailed investigation of the curing process for silica (SiO_2) coatings to determine the optimal conditions, including drying temperature and duration, for preparing silica coatings on zinc substrates using the sol-gel method.

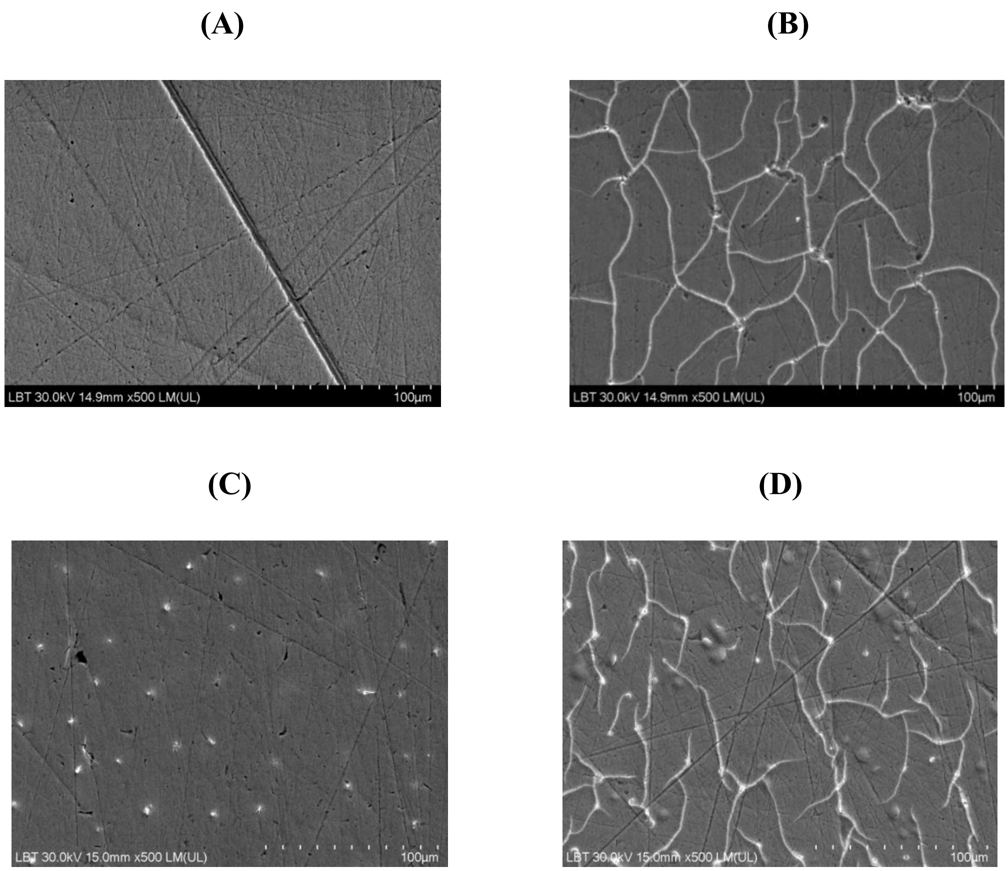


Figure 1. SEM images of silica coated Zn samples (**A**) 150 °C, 1 hour after layer deposition, (**B**) 350 °C, 1 hour after layer deposition; (**C**) 150 °C, 1 hour and (**D**) 350 °C, 1 hour after 2-weeks corrosion in 0.2 g/L Na_2SO_4 solution (pH 5).

The preparation of thin sol-gel silica coatings on zinc substrates was optimized to enhance their anti-corrosion properties. Results have shown to be very promising and led to the conclusion that the SiO_2 coatings have better protection properties when dried at 150 °C for 1 hour. Under these conditions, a compact, crack-free, and very good coverage of the Zn surface can be achieved.

6. Corrosion Behavior of Zinc Coated with Composite Silica Layers Incorporating Poly(amidoamine)- modified Graphene Oxide [12]

This research focuses on a comprehensive investigation of composite silica coatings incorporating graphene oxide modified with Poly(amidoamine) (PAMAM), primarily through electrochemical methods and morpho-structural characterizations. It compares the performance of the new silica-based coatings with those containing graphene oxide (GO), reduced graphene oxide (rGO), and graphene oxide functionalized with 3-Aminopropyltriethoxysilan (GO-APTES), respectively.

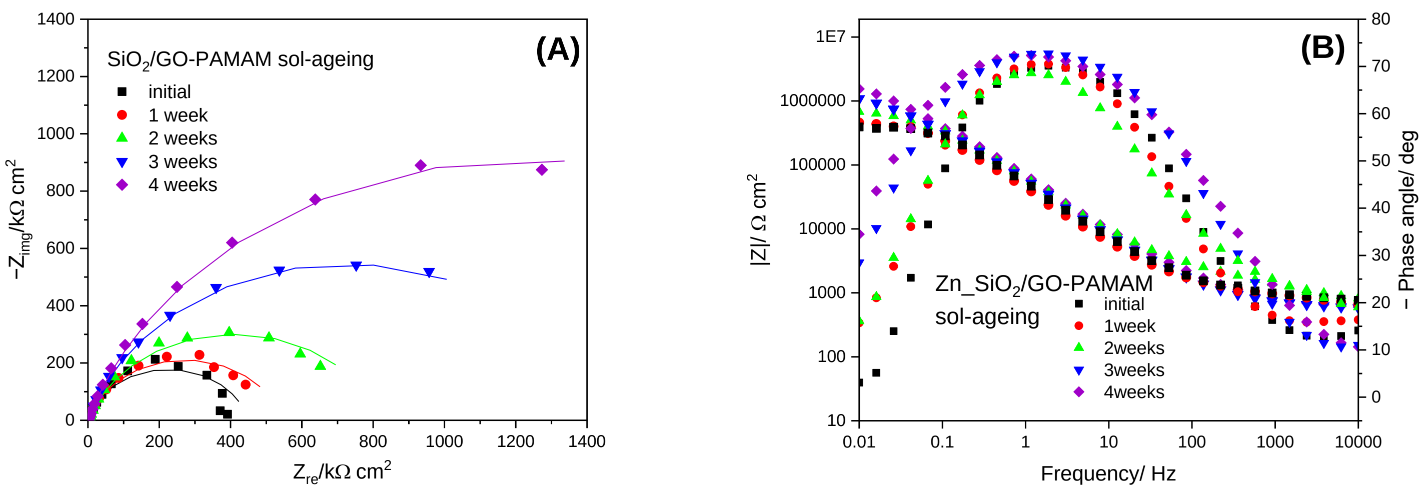
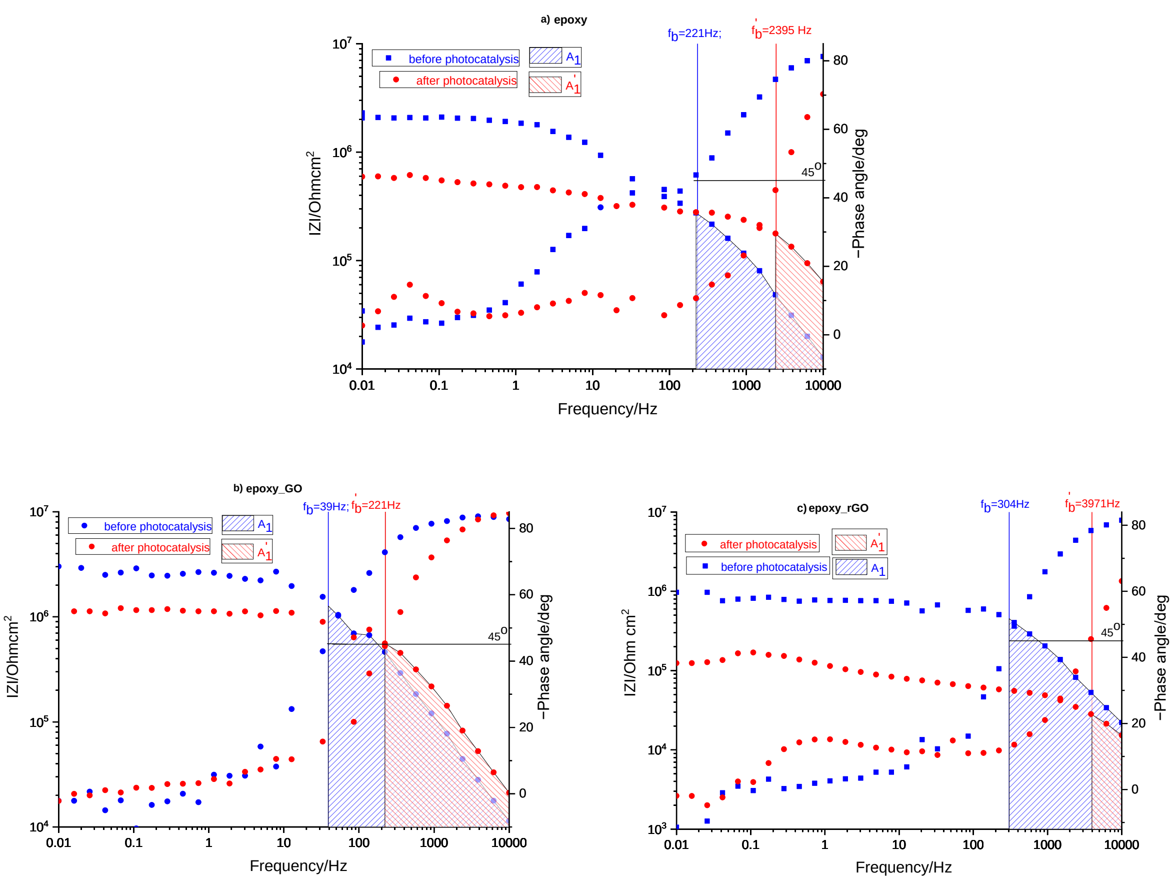


Figure 2. Nyquist (**A**) and Bode impedance spectra with phase angle plots (**B**) for SiO_2-GO-PAMAM coatings prepared with different sol-ageing duration, immersed in 0.2 g/L Na_2SO_4 solution (pH = 5.0); the solid lines represent the fitting results, and the impedance values were normalized with respect to total surface area (2 cm^2).

EIS measurements carried out in 0.2 g/L Na_2SO_4 (pH 5) allowed estimation of the different coatings' corrosion resistance. The parameters of the corrosion process were estimated by fitting the equivalent electrical circuits to the experimental diagrams for the samples prepared without sol-ageing, and after 4 weeks of sol-ageing. In all cases of composite silica coatings, the results were better than in the absence of GO-based dopants and after 4 weeks of sol-ageing (higher R_p), suggesting a favorable evolution of the polycondensation reaction. The incorporation of GO-PAMAM nanosheets into the silica matrix remarkably increased its barrier protection performance, leading to the most corrosion-resistant and water repellent coating among the investigated ones.

7. Correlations between the Anti-Corrosion Properties and the Photocatalytic Behavior of Epoxy Coatings Incorporating Modified Graphene Oxide Deposited on Zinc Substrate [13]

This work aimed to investigate the possibility of developing corrosion-resistant coatings on zinc that also function as an environmentally friendly system, utilizing the photocatalytic properties of embedded particles to degrade pollutants. Moreover, possible correlations between the photocatalytic activity of the system, composed of GO-APTES and GO-PAMAM-modified epoxy coatings on zinc substrate, and their anti-corrosion properties were investigated.



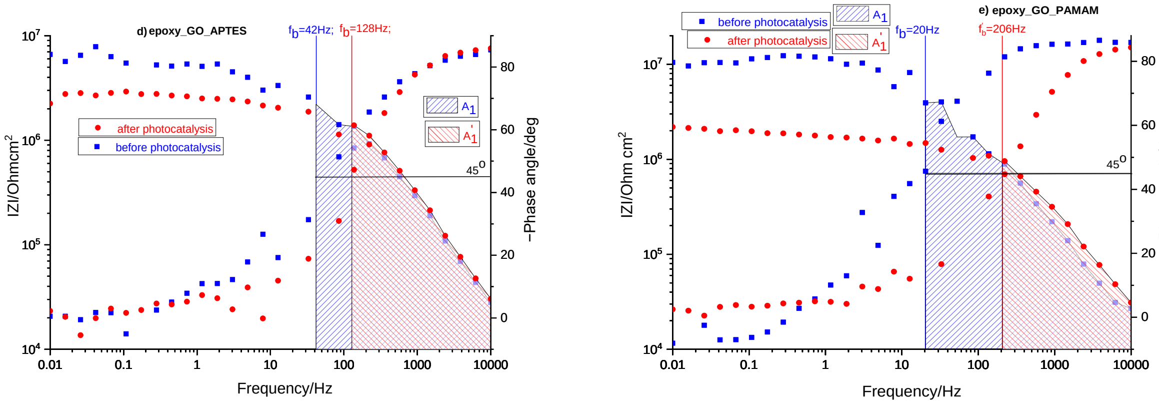


Figure 3. Comparison of Bode plots before and after photocatalysis for EP (**a**), EP-GO (**b**), EP-rGO (**c**), EP-GO-APTES (**d**), and EP-GO-PAMAM (**e**) coated zinc substrates.

Correlations between corrosion resistance and photocatalytic properties of the coatings (effect of exposure to MB and light) can be summarized as follows: before exposure, the best corrosion resistance is exhibited by Zn/EP-GO-PAMAM samples, which could be explained by their very good adhesion, due to the presence of amino groups and to its highest thickness. Furthermore, the GO-PAMAM nanosheets possess the highest degree of oxidation and the lowest electrical conductivity. The degradation of the coating by exposure to the MB solution occurs at the smallest reaction rate constant (k_{obs}), but the delamination of the coating ultimately results in a significant loss of adhesion and, consequently, reduced corrosion resistance. Low delamination despite the high photocatalytic activity was noticed for the Zn/EP-GO-APTES sample at acceptable corrosion resistance. By calculating the breakpoint frequency from EIS spectra, it was proved that the protective properties loss is due to coatings delamination during exposure to MB solution, the EP-GO-APTES still retaining the best adhesion of the coating, 98% remaining on Zn after cross-hatch test, because of the silane-functionalized GO in EP coatings, which enhances adhesion to the metal surface, forming covalent Si-O-metal bonds and increasing linkage density between the coating and the substrate.

8. Epoxy Coatings Doped with (3-Aminopropyl)triethoxysilane-Modified Silica Nanoparticles for Anti-Corrosion Protection of Zinc [14]

The study focused on two different methods for preparing (3-Aminopropyl)triethoxysilane-modified silica (SiO_2-APTES) NPs are proposed. After NP incorporation in the EP matrix, the obtained coatings prepared on zinc (Zn) substrates *via* dip coating were thoroughly characterized. The comparative study aimed to establish the optimal conditions for producing the most effective anti-corrosion coating suitable for saline environments, such as those encountered in marine transportation or during the winter season when brine solutions are frequently used.

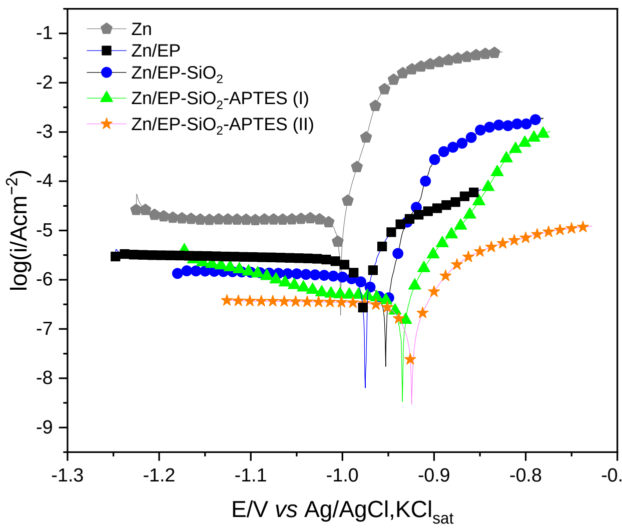


Figure 4. Polarization curves for bare Zn and coated Zn substrates recorded in 3 *wt*% NaCl solution; scan rate, 1 mV/min.

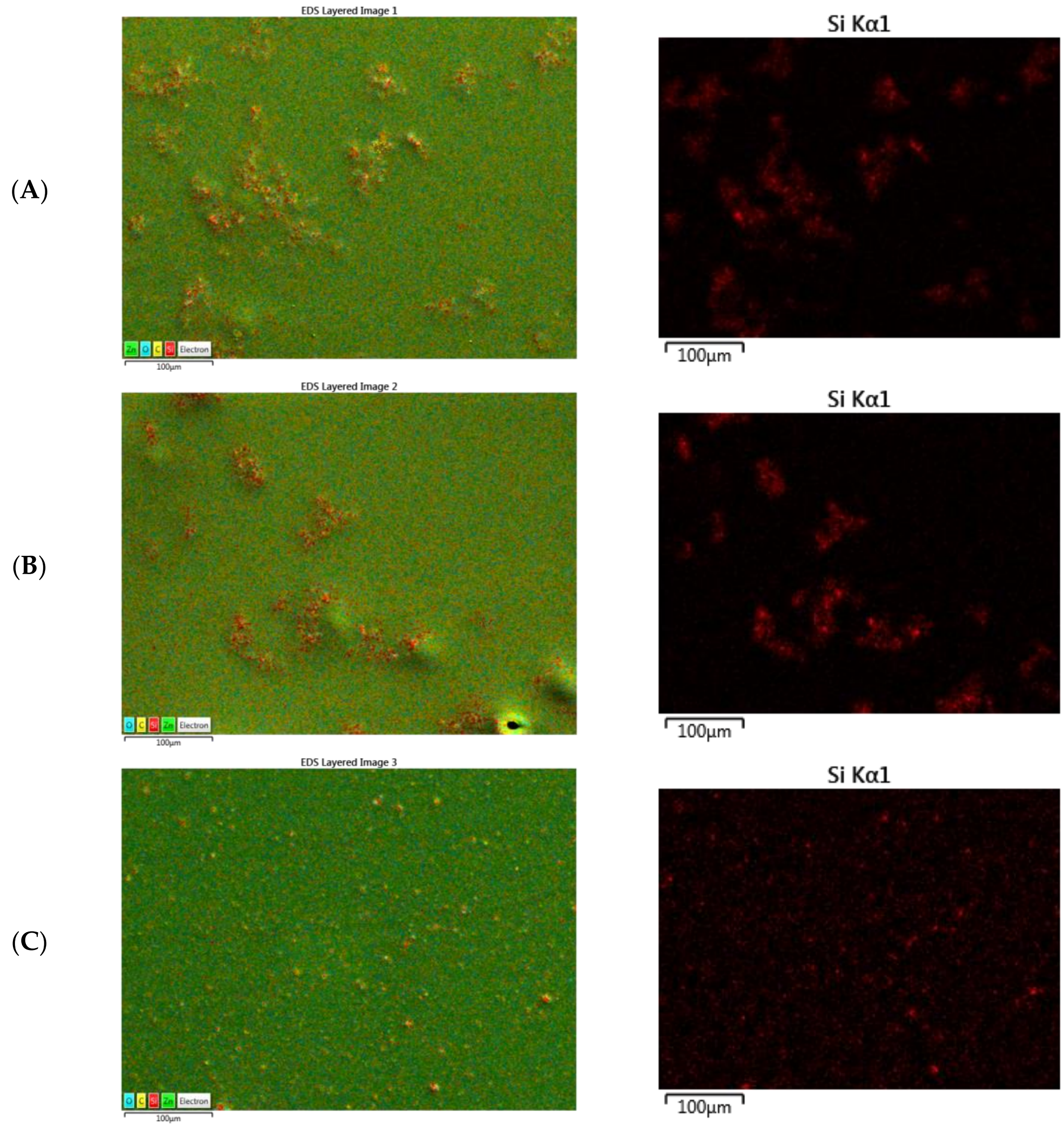
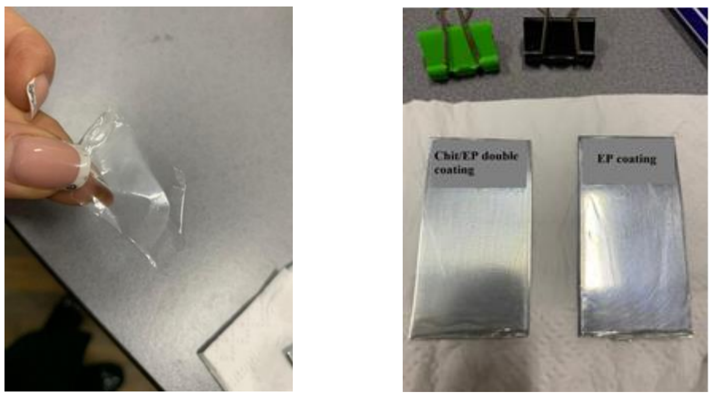


Figure 4. SEM/EDS silicon mapping of EP-SiO_2 (**A**), EP-SiO_2-APTES (I) (**B**) and EP-SiO_2-APTES (II) (**C**).

As the uniformity of the different coatings is a key element in their protective ability, the coated Zn samples were investigated *via* SEM and EDS. The SEM images of the samples (**Figure 4**) proved that by modifying the SiO_2 nanoparticles within the epoxy resin, we achieved a more uniform coating, eliminating the chance of aggregation. The repartition of the SiO_2 NPs inside the epoxy matrix was more uniform in the particular case when they were modified with APTES using method II. Modification of the SiO_2 NPs with APTES followed by their introduction into the epoxy resin (method I) led to weaker results than the deposits prepared through functionalization of the SiO_2 NPs in the epoxy gel before the addition of the hardener (method II).

9. Temporary Anti-corrosive Double Layer on Zinc Substrate Based on Chitosan Hydrogel and Epoxy Resin [15]

Chitosan/epoxy double layers were produced on zinc substrates to prepare a transparent, temporary anti-corrosive coating with enhanced protective properties in aggressive conditions, such as a marine environment. As the adhesion of the chitosan sublayer to the zinc surface can be influenced by acids, these coatings can be removable on demand after exposure to such an environment without damaging the underlying substrate [16].



(**A**) (**B**)

Figure 5. The peeled-off Chit/EP coating (**A**) and the Zn samples coated with Chit/EP and EP layer, respectively (**B**).

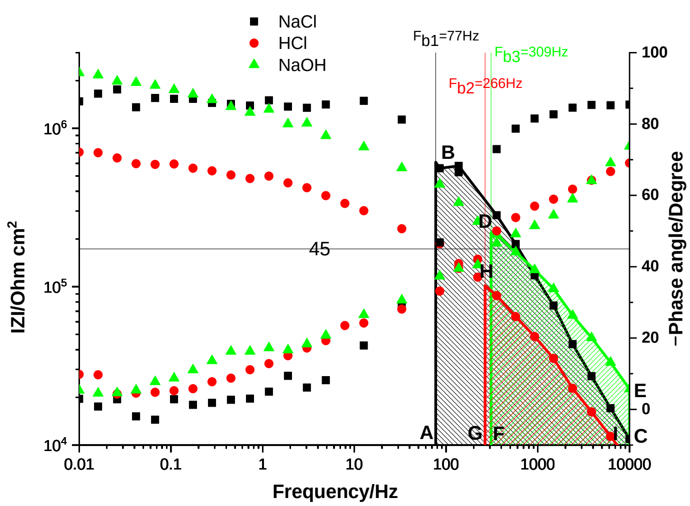


Figure 6. Bode impedance spectra of Chit/EP-coated Zn samples soaked for 2 hours in 3 *wt*% NaCl (■), 0.1 M HCl (●), and 2 M NaOH (▲) solution.

All epoxy-based coatings demonstrated excellent protective properties, with an inhibition efficiency of 99.9%. Additionally, the bilayer coatings presented the best corrosion resistance. After soaking in an acidic environment, the Chit/EP film could be peeled off on demand without damaging the underlying substrate. In conclusion, chitosan/epoxy bilayers could be a viable, eco-friendly option for producing peelable, anti-corrosive coatings suitable for transporting zinc parts, particularly in marine applications. Furthermore, the simplicity of the method should also enhance its potential for industrial application.

10. New Eco-Friendly Corrosion Inhibitor for Zinc based on Expired Detralex Drug. Adsorption and Electrochemical Studies [17]

While many studies mainly focused on single-component drugs, the present work explores a multi-component drug, highlighting the synergistic effects of its constituents on corrosion inhibition. In this context, the expired Detralex drug was investigated as a corrosion inhibitor for the zinc substrate using electrochemical tests. The adsorption type was determined following the Langmuir isotherm, and the results were corroborated with scanning electron microscopy (SEM) analysis.

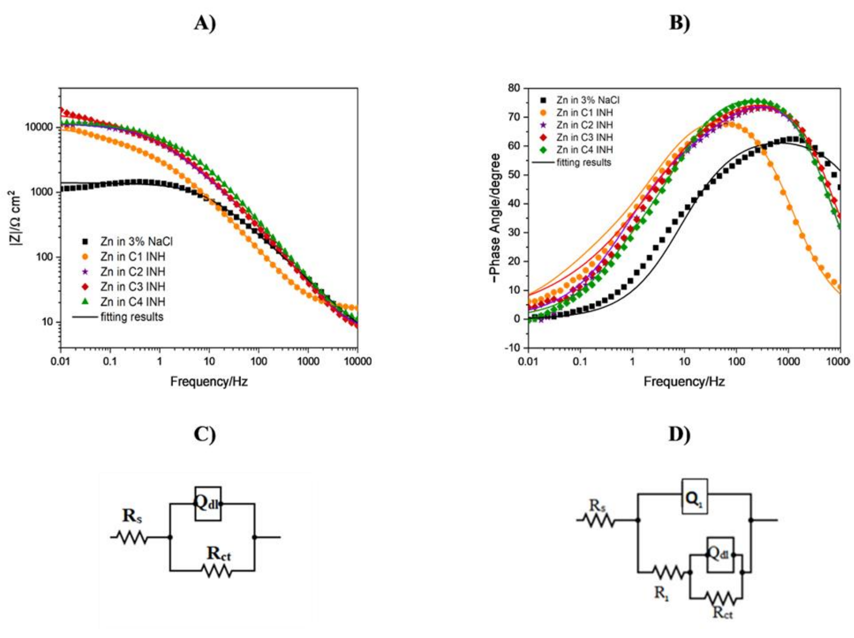


Figure 7. Bode Absolute Impedance (**A**) and Phase Angle (**B**) plots of zinc substrate immersed in 3 *wt*% NaCl containing Detralex electrolyte (pH = 6.6) at varying concentrations: 3.19×10^{-3} M (C1), 5.47×10^{-3} M (C2), 7.82×10^{-3} M (C3) and 11.43×10^{-3} M (C4). Electrical equivalent circuits for Zn in 3 *wt*% NaCl (**C**) and zinc in the presence of inhibitors (**D**).

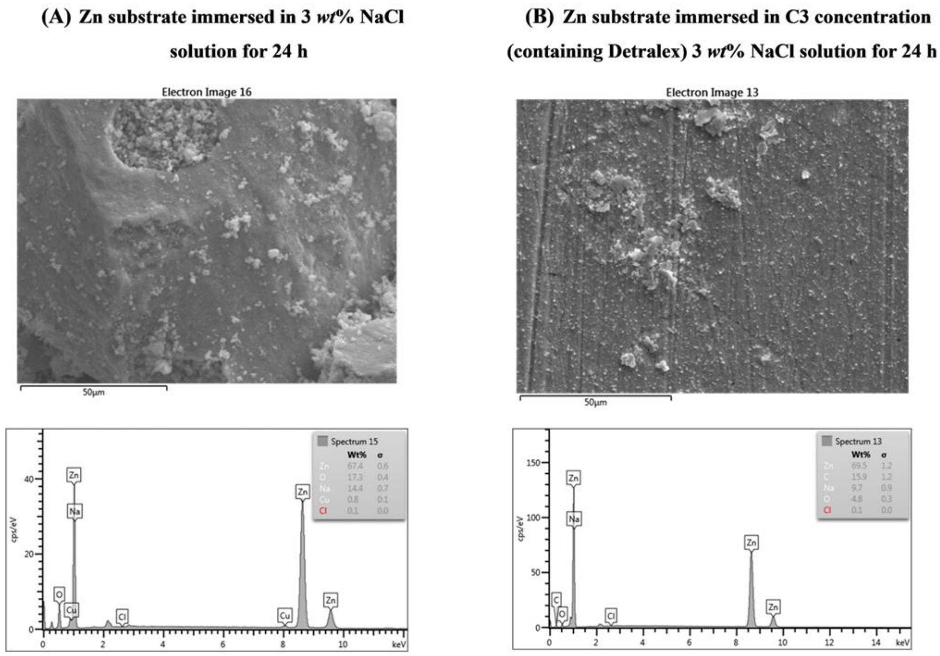


Figure 8. SEM/EDS analysis after 24-hour immersion in 3 *wt*% NaCl solution: (**A**) untreated zinc plate, (**B**) zinc plate treated with C3 inhibitor.

The equivalent electric circuits corresponding to the zinc corrosion process were determined based on EIS measurements. Without the inhibitor, the circuit presented one constant phase element and a charge transfer resistance. In the presence of Detralex, two supplementary CPE and R elements occurred and were attributed to the adsorbed protective layer [18]. The SEM results confirm that Detralex components could effectively retard the corrosion reaction of Zn in 3 *wt*% NaCl electrolyte through the adsorption of the inhibitor on the surface of the Zn electrode.

11. Summary of Research Findings

This doctoral research focused specifically on enhancing the corrosion resistance of zinc, the fourth most widely used metal globally. Zinc is renowned for its vital role in providing corrosion protection across various sectors, especially in construction and automotive manufacturing. Given its vulnerability to corrosion in harsh environments, ensuring the long-term protection of zinc is significant.

To achieve this goal, different innovative coatings were proposed:

- Silica-based coatings (with modified graphene oxides)
- Epoxy-based coatings (with modified graphene oxides/silica nanoparticles)
- Chitosan-epoxy-double layer system

To simulate real-world corrosive conditions relevant to industrial and marine applications, the selected electrolyte solutions for corrosion tests consisted of 0.2 g/L Na_2SO_4, respectively 3 *wt*% NaCl. These specific electrolytes effectively simulate atmospheric pollutants, such as sulfates, and the saline conditions typical of coastal areas, wintery roads when brine is used, or during maritime transport. Such conditions are highly aggressive and representative of those encountered by zinc components in service, especially in automotive underbody parts, marine infrastructure, and during shipping.

After preparation and investigation of the new coatings, some general conclusions can be drawn:

1. Several new **silica-based** and **epoxy-based coatings** for corrosion protection of zinc substrates were developed.
2. By incorporating nanofillers (APTES- and PAMAM-functionalized graphene oxide (GO) and APTES-modified silica (SiO_2) nanoparticles) in silica and epoxy layers, composite coatings were obtained in which the diffusion path of corrosive agents was extended, and the corrosion resistance of zinc substrate was improved.
3. The functionalization with APTES significantly improved the dispersion of both GO and silica nanoparticles within the epoxy matrix. In the case of GO, APTES enhanced the compatibility of the epoxy with the substrate and improved bonding at the filler–matrix interface, creating more insulating zones and thereby strengthening the barrier effect.
4. Similarly, PAMAM-functionalized GO improved interfacial adhesion between the epoxy coating and the zinc substrate. This strong interfacial bonding acts as an effective barrier, reducing electrolyte penetration beneath the coating and thereby delaying the onset of corrosion. A similar mechanism was observed with APTES-modified SiO_2 nanoparticles. Epoxy layers doped with these nanoparticles exhibited slower degradation, primarily due to the particles filling the micropores within the matrix. This structural densification increased the tortuosity of the diffusion path for the corrosive medium, significantly improving the coating's protective performance under immersion.

5. The zinc–epoxy–graphene oxide system demonstrated photocatalytic activity. This feature is particularly beneficial, as it enables the degradation of organic pollutants on the coating surface when exposed to light, thereby contributing to its self-cleaning properties. Such functionality is highly advantageous for outdoor or industrial applications, where surface contamination can compromise aesthetics and performance.
6. A temporary peelable coating consisting of a **chitosan-epoxy double-layer system** was developed. The removable nature of these coatings is beneficial for components that will undergo further assembly. A chitosan primer was utilized, leveraging its swelling properties in acidic environments to facilitate on-demand removal, while an epoxy top layer offered corrosion resistance under neutral conditions. The resulting system effectively protected the zinc substrate in a 3 *wt*% NaCl environment and could be cleanly removed in acidic conditions, preserving the integrity of the substrate. Such coatings are particularly advantageous for storing and transporting zinc or galvanized steel components, especially in maritime shipping, where the risk of corrosion is heightened.
7. This research investigated the corrosion inhibition properties of expired Detralex, a pharmaceutical waste product, as a sustainable and coating-free alternative for anti-corrosion. Electrochemical studies have demonstrated its effectiveness, showcasing the dual environmental and economic benefits of repurposing pharmaceutical waste as a green corrosion inhibitor.

In conclusion, this study contributes to the advancement of multifunctional protective systems for zinc, offering improved corrosion resistance, photocatalytic behavior, and sustainable strategies for temporary protection and the reuse of pharmaceutical waste. These findings broaden the application potential of zinc-based components and align with growing environmental and industrial demands for smarter, more adaptable protection technologies.

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