Research on enhancing the corrosion resistance of epoxy-modified Fe_3O_4 coating on steel in a 3.5% NaCl environment

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ABSTRACT

This study aims to improve the corrosion resistance of steel in a 3.5% NaCl environment by developing epoxy coatings containing surface-modified Fe_3O_4 nanoparticles. Fe_3O_4 was synthesized via co-precipitation, functionalized with 3-aminopropyltriethoxysilane (APTES) to improve dispersion, and combined with triethanolamine (TEA) as a surfactant to stabilize morphology. Five coating systems, pure epoxy, epoxy–Fe_3O_4, epoxy–Fe_3O_4/TEA, epoxy–APTES–Fe_3O_4, and epoxy–APTES–Fe_3O_4/TEA, were prepared. The nanoparticle structure and morphology were analyzed using XRD, FT-IR, SEM and EDX, while corrosion resistance was evaluated via potentiodynamic polarization (PDP) và electrochemical impedance spectroscopy (EIS). Results showed that APTES functionalization improved nanoparticle dispersion and interfacial bonding, while TEA reduced particle size and prevented agglomeration. The epoxy–APTES–Fe_3O_4/TEA system exhibited the best corrosion protection. These findings highlight the potential of modified epoxy nanocomposite coatings for marine steel protection.

Keywords: Nanopigment; APTES; Epoxy coating; Corrosion resistance; Tafel.

1. INTRODUCTION

Epoxy coatings are widely used for steel corrosion protection due to their strong adhesion, high mechanical strength, and chemical resistance, acting as barriers against corrosive ions. However, microdefects in the crosslinked network after curing can allow ion penetration, making steel highly susceptible to corrosion, especially in marine or NaCl environments, where redox reactions and rust formation degrade performance. To enhance protection, nanoparticles such as ZnO [1] and SiO_2 [2] have been incorporated into epoxy to improve barrier properties. Magnetic (Fe_3O_4) nanoparticles are particularly attractive for their magnetic properties, strong bonding with epoxy, and uniform dispersion, with applications in drug delivery, MRI, biosensors, and data storage [3]. Their physicochemical properties depend heavily on the synthesis method; among various routes, the co-precipitation method–used in this study–is favored for its simplicity, low cost, and scalability [4].

Nevertheless, most nanoparticles are inherently incompatible with epoxy coatings due to differences in polarity, absence of chemical bonding, and a tendency to agglomerate. To overcome this, nanoparticle surfaces are often functionalized using coupling agents such as silanes or fatty acids. Silanization is among the most widely adopted methods for modifying nanoparticle polarity, improving dispersion, enhancing adhesion, and stabilizing coatings. M.J. Palimi et al. [5] used 3-aminopropyltrimethoxysilane (APTMS) to modify Cr_2O_3 nanoparticles, improving their dispersion in polyurethane and enhancing corrosion resistance. Park et al. [6] functionalized Fe_3O_4 with APTES, which improved Fe_3O_4 dispersion in epoxy resins, increased composite

magnetization by 110%, and reduced wear loss by 60%. Therefore, functionalization of Fe_3O_4 surfaces is essential to enhance dispersion and compatibility with epoxy. APTES is a widely used silane coupling agent for modifying metal oxide nanoparticles, improving compatibility and dispersion through $–NH_2$ groups that can react with epoxy, thereby preventing agglomeration and significantly enhancing coating performance.

In addition to surface functionalization, TEA acts as a nonionic surfactant that stabilizes Fe_3O_4 dispersion and controls particle morphology. Javidparvar et al. [7] reported that TEA produces nanoparticles with diverse morphologies, improves epoxy dispersion and particle–matrix interaction, reduces surface energy, enhances hydrophilicity and compatibility, prevents agglomeration, and results in uniform, smooth coatings with superior corrosion resistance.

Although metal oxide–epoxy nanocomposite coatings have been studied previously, the combination of APTES-functionalized Fe_3O_4 with TEA surfactant in epoxy coatings for steel corrosion protection in 3.5% NaCl environments has not yet been investigated. For these reasons, the present work holds both scientific and practical significance, aiming to develop effective, environmentally friendly coatings that contribute to sustainable corrosion protection for steel structures in marine environments.

2. EXPERIMENT

2.1. Materials

$FeCl_3.6H_2O$, $FeCl_2.4H_2O$ were purchased from Merck (Germany). NaOH (≥ 99%), TEA (≥ 98%), HCl (30–34%), absolute ethanol (EtOH, ≥99.5%), and APTES (≥ 99%) were purchased from Xilong (China). A commercial waterborne epoxy resin was employed as the coating matrix. Mild steel plates (φ = 90 mm) were used as substrates.

2.2. Experimental

2.2.1. Research methodologies

The crystalline phases of the nanopigments were characterized by XRD (Aeris, Malvern Panalytical) in a 2θ range of 5° - 80°. Particle size, morphology, and elemental composition were analyzed using SEM (MIRA MLU, TESCAN) with EDX. FTIR (Platinum ATR Alpha II, Bruker, Germany, 400 - 4000 cm^{-1}) was used to confirm APTES grafting on Fe_3O_4 surfaces. Corrosion resistance of epoxy coatings with unmodified and modified Fe_3O_4 was assessed by potentiodynamic polarization in a three–electrode cell (coated steel working electrode, stainless steel counter electrode, Ag/AgCl reference electrode) in 3.5% NaCl, with data processed using Nova 2.0 software.

2.2.2. Synthesis of Fe_3O_4 nanoparticles

Fe_3O_4 nanoparticles were synthesized via a co-precipitation route adapted from Ganesha Antarnusa et al. [8]. 7 g $FeCl_3.6H_2O$ and 3.6 g $FeCl_2.4H_2O$ were dissolved separately in deionized water, then combined at a 2:1 molar ratio under stirring to form an orange solution. The mixture was heated to 60 °C and stirred at 450 rpm, after which 50 mL NH_4OH was added dropwise and the reaction was maintained for 90 min, leading to the formation of a black Fe_3O_4 suspension. The precipitate was magnetically separated, washed repeatedly with deionized water until no NH_4OH odor remained, dried at 60 °C for 24 h, and ground to obtain fine Fe_3O_4 nanopowder.

2.2.3. Synthesis of Fe_3O_4/TEA nanoparticle

Fe_3O_4/TEA nanoparticles were prepared following the complex co-precipitation method reported by Tian Xia et al. [9]. $FeCl_3.6H_2O$ (0.3599 g), $FeCl_2.4H_2O$ (0.3336 g), TEA (0.8951 g), and NaOH (1.44 g) were individually dissolved in distilled water under magnetic stirring. NaOH and TEA solutions were then added to the $FeCl_3$ and $FeCl_2$ solutions with stirring for 5 min, respectively, to generate FeOOH colloids and Fe(II)–TEA complexes. The resulting mixtures were

rapidly combined, transferred to a PTFE beaker, sealed, and heated at 100 °C for 4 h. After natural cooling to room temperature, the product was washed repeatedly with deionized water and dried at 75 °C for 12 h.

2.2.4. Silane modification of Fe_3O_4 nanoparticles

Surface modification with APTES was performed according to A.A. Javidparvar et al. [7]. 0.5 g nanomagnetite, 95.3 g of EtOH, 3.8 g of distilled water, and 0.3 g of APTES were placed under a homogenizer. Hydrochloric acid was added dropwise to the suspension to adjust the pH to 1.5 – 2, providing a proper condition for silane hydrolysis. The suspension was kept at 60 °C for 1 h. For the silane condensation, the pH of the solution was adjusted in the range of 8–10, and the suspension was stirred for 2 h. Finally, the mixture was centrifuged and washed five times with EtOH and deionized water and then dried in an oven at 80 °C.

2.2.5. Epoxy–nanocomposites preparation

Epoxy nanocomposites were prepared by the addition of 1 wt.% of unmodified or modified Fe_3O_4 nanoparticles to 30 g of waterborne epoxy resin (with solid content 48±2wt.%). First, nanoparticles were mechanically mixed with the epoxy resin, and then ultrasonication was performed for 5 min to ensure homogeneous dispersion. The mixture was then spray–coated onto pre–cleaned steel substrates. Coatings were air–dried under ambient conditions, and the dry film thickness of the coating was 20 - 30 µm.

3. RESULTS AND DISCUSSION

3.1. Nanopigment phase composition and morphology evaluation

The XRD patterns (figure 1) of Fe_3O_4 and APTES–Fe_3O_4/TEA samples are consistent with the standard spinel Fe_3O_4 structure (JCPDS 00-065-0731), showing diffraction peaks at 30.13°, 35.53°, 43.17°, 53.66°, 57.16°, and 62.77°, corresponding to the (220), (311), (400), (422), (511), and (440) planes [10]. The peak positions remain unchanged; however, the peak intensity of Fe_3O_4 is higher than that of APTES–Fe_3O_4/TEA, indicating that the surfactant reduced the crystallinity of the nanoparticles.

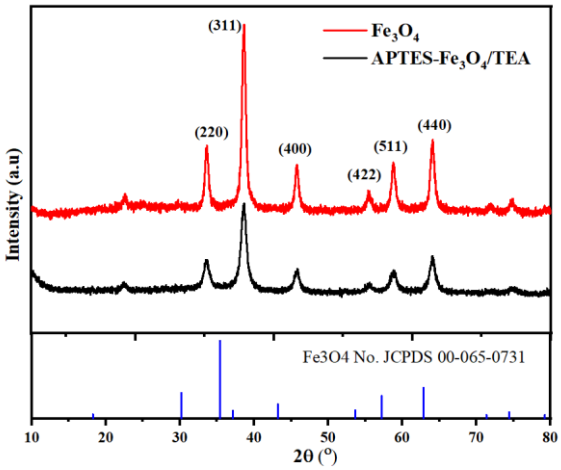

Figure 1. *X-ray diffraction patterns for Fe_3O_4 and APTES-Fe_3O_4/TEA.*

The crystal size of nanoparticles was calculated by the Scherrer equation:

$$D = \frac{K \times \lambda}{B \times \cos(\theta)} \quad (1)$$

Where D is the crystallite size (nm), λ is the Cu-K_α wavelength (0,154 nm), and θ is the Bragg angle of the most intense peak. β is the full width at half maximum (FWHM) in radians.

Results showed that the crystal size of Fe_3O_4 and APTES–Fe_3O_4/TEA was about 23.57 nm and 14.05 nm, respectively. The reduction in crystallite size for the modified sample indicates that APTES and TEA act as capping agents, inhibiting crystal growth and producing smaller nanoparticles. This observation is consistent with previous studies [11].

The surface morphology of Fe_3O_4 and APTES–Fe_3O_4/TEA nanopigments was examined via SEM (figure 2). The Fe_3O_4 nanoparticles are spherical, with an average particle size of ~30 nm, although partial agglomeration is observed. After APTES modification, the spherical morphology is retained, but slight interparticle crosslinking is visible. The average particle diameter increases to ~33 nm, attributable to the silane coating layer. Notably, the particle size from SEM is larger than the crystallite size from XRD, as individual particles consist of multiple crystallites aggregated together [12].

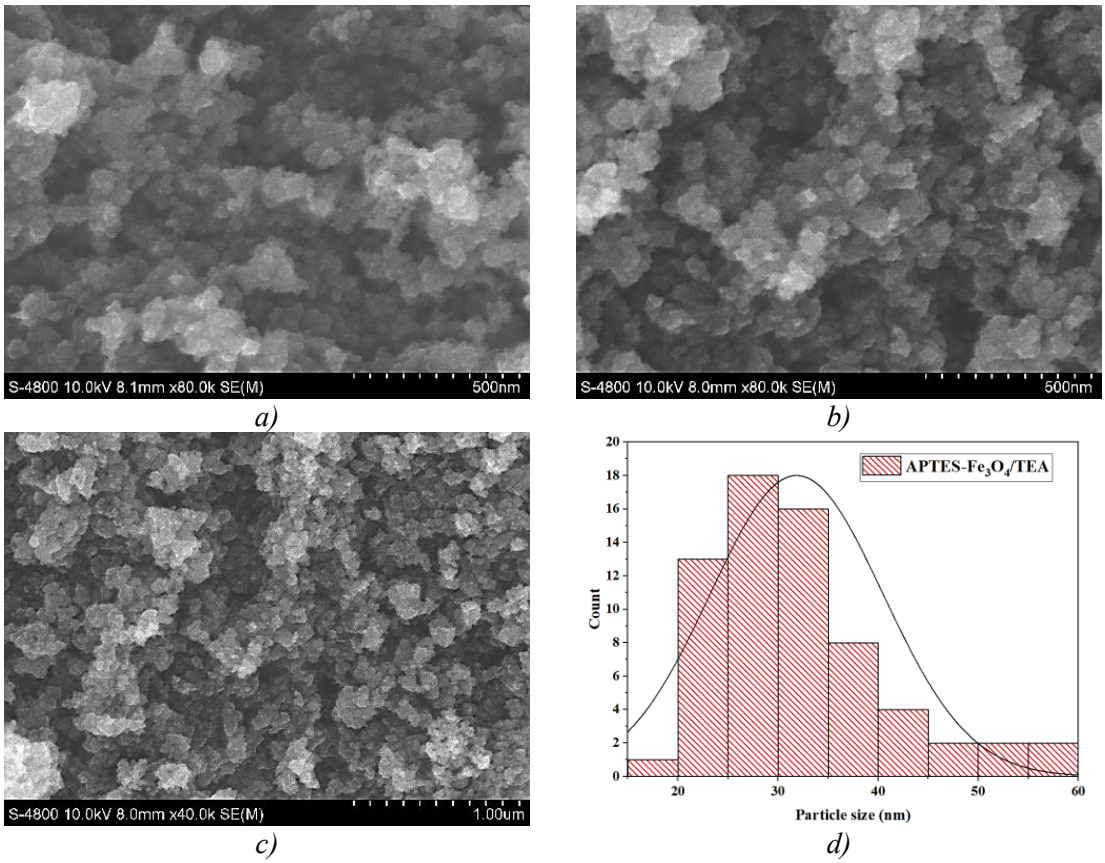

Figure 2. *a) SEM images of Fe_3O_4; b), c) SEM images of APTES-Fe_3O_4/TEA (500 nm and 1000 µm resolution; d) the particle size distribution of APTES-Fe_3O_4/TEA.*

3.2. Energy-dispersive X-ray spectroscopy (EDX)

The elemental composition of APTES–Fe_3O_4/TEA nanoparticles, as determined by EDX analysis and shown in figure 3, reveals the presence of all expected elements: Fe, Si, O, N, and C, consistent with the proposed structure. These findings are in agreement with previous studies [13, 14]. This confirms the good dispersion of the nanoparticles, demonstrating that the synthesis method employed is both effective and efficient in preserving the desired elemental composition during the preparation of Fe_3O_4 nanoparticles. The presence of Si and N elements indicates the incorporation of APTES and TEA. These results suggest that APTES formed a silane coating layer through Si–O–Fe covalent linkages with the nanoparticle surface, and that TEA molecules are complexed onto this silane layer.

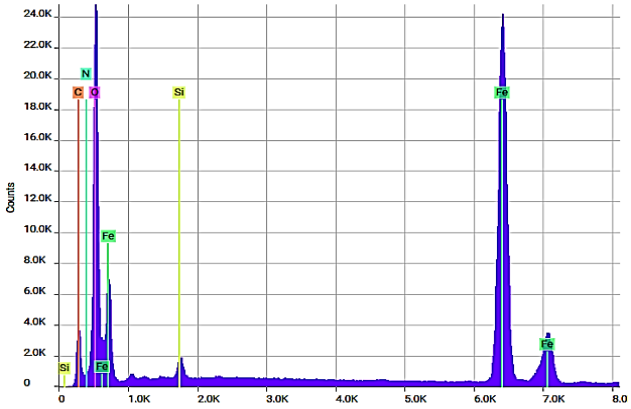

Figure 3. *EDX elemental analysis of APTES-Fe_3O_4/TEA.*

3.3. FT-IR analysis

FTIR spectra (figure 4) indicate that APTES–modified Fe_3O_4 retains the main features of unmodified Fe_3O_4 but shows higher overall absorbance and an additional peak at 1010 cm^{-1}, corresponding to Si–O–Si stretching, confirming successful APTES adsorption on the nanoparticle surface. Increased absorbance is also observed at 3422 cm^{-1}, 1627 cm^{-1}, and 629 cm^{-1}. The covalent Fe–O–Si linkage is evidenced by absorption bands at 629 cm^{-1} and 418 cm^{-1}, though these overlap with Fe–O vibrations, making them difficult to distinguish. Notably, the modified sample exhibits broader peaks around 629 cm^{-1} compared to the unmodified one. Additionally, peaks at 3448 cm^{-1} and 1627 cm^{-1} correspond to Fe–OH bending, –OH stretching, and N–H stretching vibrations, further supporting APTES grafting. These results are consistent with previous reports [15].

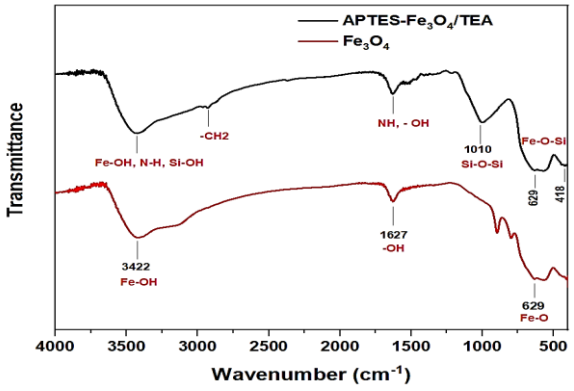

Figure 4. *FT-IR spectrum of Fe_3O_4 and APTES–Fe_3O_4/TEA.*

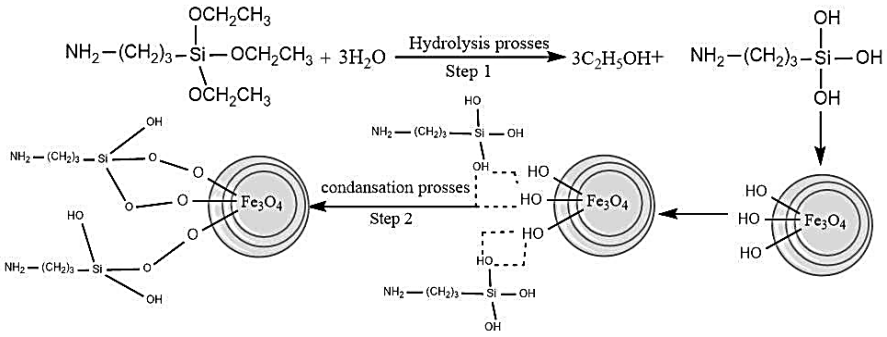

Figure 5. *A schematic of grafting of APTES on the Fe_3O_4 nanoparticles surface.*

APTES was successfully grafted on the surface of Fe_3O_4 nanopigments through chemical bonding, resulting in Fe–O–Si bond formation, as shown in figure 5.

3.4. Potentiodynamic polarization (PDP)

Incorporating 1 wt.% nanopigments into epoxy coatings and their effect on corrosion protection were studied using the PDP test [16, 17]. Figure 6 presents the Tafel plots of pure epoxy, epoxy–Fe_3O_4, epoxy–Fe_3O_4/TEA, epoxy–APTES–Fe_3O_4, and epoxy–APTES–Fe_3O_4/TEA coatings in 3.5% NaCl solution. The i_{corr} and E_{corr} values for all samples are summarized in table 1. As presented in table 1, the pure epoxy exhibited the lowest protective efficiency, with a high i_{corr} (10^{-3} - 1.58×10^{-3} mA/cm^2) and a pronounced negative shift in E_{corr} (- 0.91 V after 21 days), indicating severe deterioration of the barrier properties.

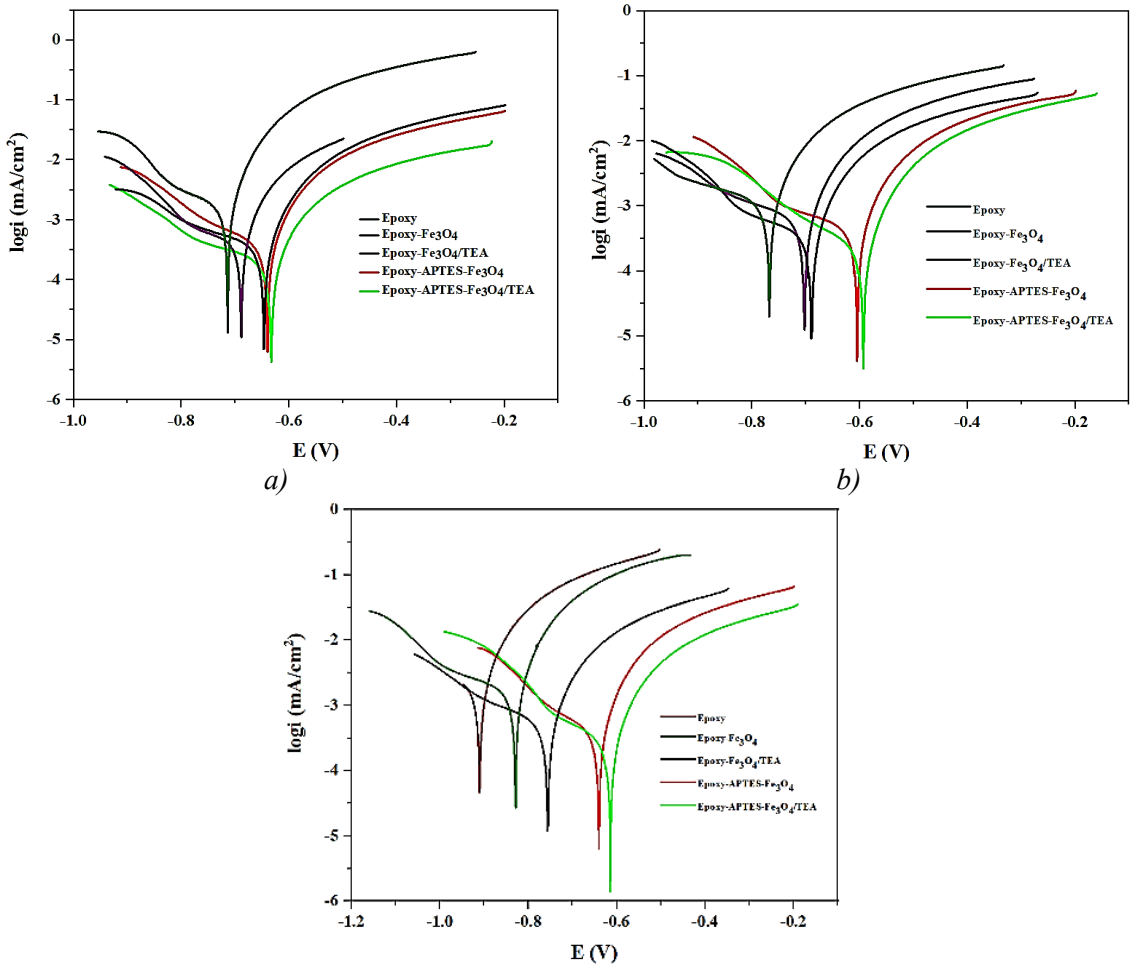

Figure 6. *Potentiodynamic polarization curves of nanocomposite coatings in 3.5% NaCl solution after a) 1 day, b) 7 days and 21 days of immersion.*

In comparison, coatings incorporating unmodified Fe_3O_4 nanoparticles (with or without TEA) initially improved corrosion resistance, but their performance declined significantly upon prolonged immersion. This degradation is attributed to nanoparticle agglomeration, which introduces micro-defects and facilitates electrolyte ingress into the polymer matrix. By contrast, coatings containing APTES-functionalized Fe_3O_4 nanoparticles exhibited markedly enhanced corrosion resistance. Both epoxy–APTES–Fe_3O_4 and epoxy–APTES–Fe_3O_4/TEA showed more positive E_{corr} values and significantly reduced i_{corr} compared to other samples. Notably, epoxy–

APTES–Fe_3O_4/TEA maintained the lowest i_{corr} (1.26×10^{-4} mA/cm^2 after 21 days), confirming superior long-term electrochemical stability and effective suppression of chloride ion ingress. This mechanism arises from the synergistic effect of the $–NH_2$ groups in APTES, which promote stronger cross-linking of the epoxy coating through reactions with the glycidyl groups of the epoxy resin, resulting in a denser network structure and improved barrier properties. In addition, the incorporation of TEA reduces nanoparticle size and ensures uniform dispersion, thereby providing optimal corrosion protection.

Table 1. *Electrochemical parameters of coatings derived from Tafel extrapolation.*

Immersion time	1 day		7 days		21 days	
Sample name	E_{corr} (V)	i_{corr} (mA/cm^2)	E_{corr} (V)	i_{corr} (mA/cm^2)	E_{corr} (V)	i_{corr} (mA/cm^2)
Pure epoxy	-0.71	10^{-3}	-0.77	1.25×10^{-3}	-0.91	1.58×10^{-3}
Epoxy–Fe_3O_4	-0.69	3.16×10^{-4}	-0.70	6.31×10^{-4}	-0.83	7.94×10^{-4}
Epoxy–Fe_3O_4/TEA	-0.65	2.51×10^{-4}	-0.69	3.16×10^{-4}	-0.76	3.89×10^{-4}
Epoxy–APTES–Fe_3O_4	-0.64	2.41×10^{-4}	-0.60	2.29×10^{-4}	-0.62	2.39×10^{-4}
Epoxy–APTES–Fe_3O_4/TEA	-0.63	1.58×10^{-4}	-0.59	1.25×10^{-4}	-0.61	1.26×10^{-4}

3.5. Electrochemical impedance spectroscopy (EIS)

The corrosion protection performance of the epoxy coatings was further evaluated by EIS and the results are presented in the Bode plots in figure 7. Organic coatings represent a complex system that is often modeled only hypothetically and may not accurately reflect the actual mechanisms due to the simultaneous occurrence of multiple processes. Therefore, determining an appropriate equivalent electrical circuit model for Nyquist plots without introducing errors is extremely challenging. Bode plots–providing impedance values at low frequencies and phase angles at high frequencies–are considered sufficient to evaluate the corrosion resistance of the coatings.

As shown in figure 7, the pure epoxy coating exhibited a pronounced decrease in |Z| from day 1 to day 21, accompanied by a significant negative shift in phase angle, indicating water uptake and the loss of protective ability over immersion time. For the epoxy–Fe_3O_4 and epoxy–Fe_3O_4/TEA samples, improved protection was observed in the early stage compared to pure epoxy; however, both |Z| and phase angle decreased significantly after 21 days due to nanoparticle agglomeration, which facilitated electrolyte penetration. In contrast, the epoxy–APTES–Fe_3O_4 coating maintained higher |Z| values and more stable phase angles throughout immersion, indicating the formation of a denser barrier network resulting from the interaction between APTES and the epoxy matrix. Notably, the epoxy–APTES–Fe_3O_4/TEA coating exhibited the best corrosion protection, with the highest |Z| at low frequency and the smallest reduction in phase angle at high frequency, confirming its superior barrier properties.

The incorporation of APTES improved nanoparticle dispersion in the epoxy matrix, enhanced barrier efficiency, and prolonged the diffusion path of aggressive ions through the coating. Furthermore, the presence of TEA effectively contributed to corrosion inhibition. TEA acted as a surface-capping agent, reducing the particle size of the nano-pigments. This reduction in pigment size led to improved barrier performance, better nanoparticle dispersion within the epoxy matrix, and minimized agglomeration, thereby enhancing the overall corrosion resistance of the coating.

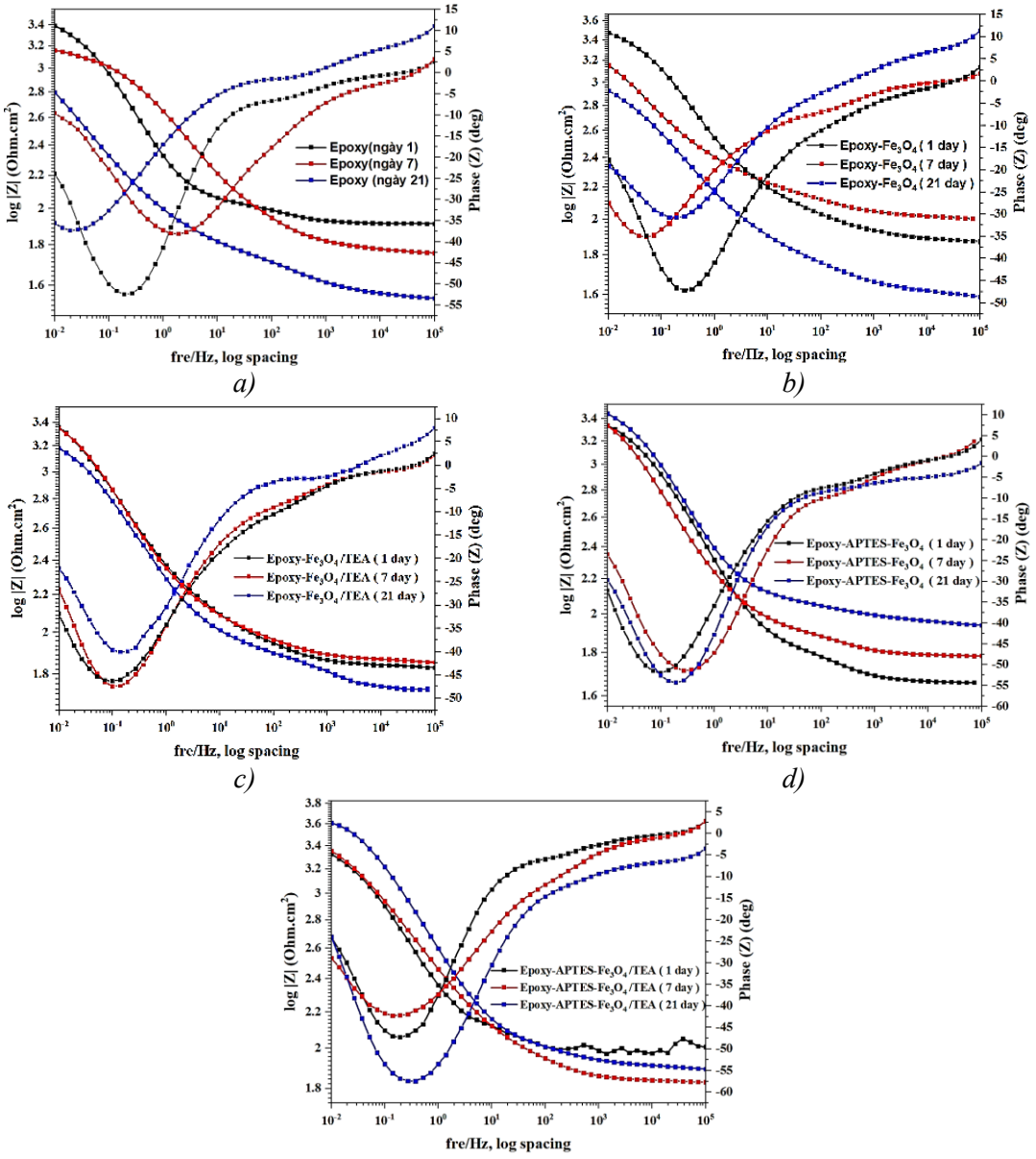

Figure 7. *Bode plots of the pure epoxy (a), epoxy–Fe_3O_4 (b), epoxy–Fe_3O_4/TEA (c), epoxy–APTES–Fe_3O_4 (d), epoxy–APTES–Fe_3O_4/TEA (e) after 1, 7 and 21 days immersion in 3.5 wt.% NaCl solutions.*

4. CONCLUSIONS

This study successfully synthesized Fe_3O_4 nanoparticles surface-modified with APTES, while employing TEA as a surfactant to improve dispersion. XRD, SEM, FTIR, and EDX analyses confirmed that the crystal structure, chemical composition, and the presence of functional groups on the nanoparticle surface were consistent with the research objectives. Electrochemical results (PDP, EIS) showed that epoxy–APTES–Fe_3O_4/TEA samples achieved the lowest i_{corr}, most positive E_{corr}, and highest impedance, demonstrating superior long-term corrosion resistance. The combination of APTES and TEA enhanced the barrier performance of the coating, offering promising potential for applications in protecting steel structures in marine environments.

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TÓM TẮT

Nghiên cứu nâng cao tính chất chống ăn mòn thép trong môi trường NaCl 3,5% của lớp phủ epoxy-Fe_3O_4 biến tính

Nghiên cứu này tập trung nâng cao khả năng chống ăn mòn của thép trong dung dịch NaCl 3,5% thông qua việc chế tạo lớp phủ epoxy chứa hạt nano Fe_3O_4 biến tính. Nano Fe_3O_4 được tổng hợp bằng phương pháp đồng kết tủa và biến tính bằng APTES để cải thiện khả năng phân tán và tương thích với nền epoxy. TEA được sử dụng như chất hoạt động bề mặt nhằm ổn định hình thái hạt và hỗ trợ phân tán đồng đều trong lớp phủ. Năm mẫu được chế tạo gồm: epoxy nguyên chất, epoxy–Fe_3O_4/TEA, epoxy–APTES–Fe_3O_4 và epoxy–APTES–Fe_3O_4/TEA. Cấu trúc và hình thái hạt nano được đặc trưng bằng XRD, FTIR, SEM và EDX. Khả năng chống ăn mòn được khảo sát bằng PDP và EIS. Kết quả cho thấy việc biến tính bằng APTES và TEA giúp giảm i_{corr}, dịch chuyển E_{corr} về dương và giá trị $|Z|$ ở tần số thấp cao nhất, chứng tỏ hiệu quả bảo vệ cao hơn, hứa hẹn ứng dụng trong bảo vệ kết cấu thép trong môi trường biển, kéo dài tuổi thọ và giảm chi phí bảo trì.

Từ khoá: Nanopigment; APTES; Lớp phủ epoxy; Khả năng chống ăn mòn; Tafel.