FABRICATION OF ORGANIC SILANE-MODIFIED GRAPHENE OXIDE COATINGS FOR CORROSION PROTECTION OF METALS

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ABSTRACT

This study presents the synthesis of graphene oxide (GO) functionalized with organic silane compounds, namely 3-glycidoxypropyltrimethoxysilane (GPTES) and N-(2-aminoethyl)-3-aminopropyltriethoxysilane (KH-602), dispersed in an epoxy matrix to enhance the corrosion protection of metals. The morphological and structural characteristics of the materials were comprehensively analyzed using advanced physicochemical techniques, including Fourier transform infrared spectroscopy (FT-IR), field-emission scanning electron microscopy (FE-SEM), and energy-dispersive X-ray spectroscopy (EDX). The anticorrosion performance of the coating systems was evaluated by electrochemical impedance spectroscopy (EIS) and Tafel polarization measurements. The results demonstrate that epoxy coatings incorporating functionalized GO exhibited significantly improved corrosion resistance compared with pristine epoxy, with inhibition efficiencies above 90% under laboratory immersion conditions. This highlights their promising potential for application in protective coatings for metals in corrosive environments.

Keywords: *Graphene oxide, organic silane, corrosion protection.*

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1. INTRODUCTION

Corrosion of metals remains a critical challenge in materials science and engineering, leading to severe degradation of mechanical properties, structural instability, and substantial global economic losses. According to reports, each year, corrosion-related expenses represent about 3 - 4% of global GDP and directly impact primary industries, including infrastructure, transportation, marine exploitation, energy, and defense [1]. Conventional protection strategies, including metallic coatings, anodization, and organic polymer coatings, have provided certain benefits; however, their long-term durability remains limited, particularly under harsh environments characterized by high humidity, chloride ions, or fluctuating temperatures. Therefore, developing advanced protective coatings that can effectively prevent corrosive species while maintaining long-term mechanical stability is essential to extending the service life of metallic materials.

In recent years, graphene oxide (GO) has attracted considerable attention in the field of anticorrosive coatings because of its unique two-dimensional structure, extensive specific surface area, and abundant oxygen-containing functional groups [2]. When incorporated into polymer matrices, particularly epoxy resins, GO nanosheets can extend the diffusion pathways, significantly reducing the transport rate of water, oxygen, and chloride ions to the metal surface. In addition, GO contributes to the mechanical reinforcement of the coating, enabling epoxy–GO composites to exhibit superior corrosion resistance compared to pristine epoxy. However, the direct use of GO also presents several limitations. Due to its hydrophilic nature and strong Van der Waals interactions, GO sheets readily agglomerate, which prevents their uniform dispersion in epoxy matrices and weakens the formation of stable interfacial bonding, thereby limiting the protective performance of the coating and increasing the risk of delamination under aggressive environmental conditions. Therefore, surface functionalization of GO is essential to enhance its dispersibility and improve compatibility with epoxy resins [3].

Among various modification strategies, using organic silane compounds is considered an effective and practical approach. Silanes such as 3-glycidoxypropyltrimethoxysilane (GPTES), N-(2-

aminoethyl)-3-aminopropyltriethoxysilane (KH-602) undergo hydrolysis-condensation of their alkoxysilane groups, forming covalent Si-O-C linkages with hydroxyl groups on the GO surface while simultaneously introducing organic functionalities compatible with epoxy resins. Specifically, GPTES carries an epoxy group capable of ring-opening reactions with –OH or –COOH groups of GO as well as with epoxy curing agents, whereas KH602 contains diamine groups that not only graft onto GO but also react with epoxy during curing, thereby establishing a tightly cross-linked and durable covalent network [4].

In this study, silane-modified GO was incorporated into epoxy matrices to enhance filler dispersion, reduce micropore formation, and construct a more stable barrier structure. The work aims to evaluate the structural and anticorrosive properties of epoxy/silane–GO composite coatings applied to CT3 steel substrates.

2. MATERIALS AND METHODS

2.1. Materials

- GO was prepared following a previously published procedure [5].

- 3-glycidoxypropyltrimethoxysilane (GPTES), N-(2-aminoethyl)-3-aminopropyltri-ethoxysilane (KH-602) were of analytical grade and obtained from Merck. Chemical structures of GPTES and KH-602 are presented in Figure 1.

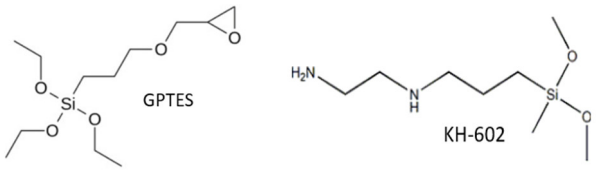


Figure 1. Chemical structures of GPTES and KH-602

- Methyl isobutyl ketone (Macklin, China) was employed as the solvent in paint formulation.

- Ethanol, methanol, dibutyltin dilaurate, and N, N-dimethylformamide (DMF) and sodium chloride (NaCl), also of analytical grade, were procured from Macklin (China).

- Epoxy resin YD011-X75 (solid content 75%) and the polyamide curing agent G700-X60 were supplied by Kukdo Chemical Co., Ltd. (South Korea).

Mild steel (CT3 grade) is the metallic substrate for electrode preparation and corrosion testing. Before use, the steel surfaces were mechanically polished in sequence with abrasive papers of 180, 400, 1000, and 2000 grit sizes.

All chemicals and reagents were employed as received without any additional purification.

2.2. Synthesis of organic silane functionalized graphene oxide

Firstly, 2.5g of GO was ultrasonically dispersed in a mixed solvent of 25mL DMF and 25mL ethanol using a 1500W probe for 5 hours to achieve thorough exfoliation and homogeneous dispersion. The suspension obtained was homogenized at high speed for 3 hours. Then, GPTES was introduced into the mixture at a ratio of 1mL per gram of GO, together with 50mL of ethanol. To promote the reaction, 0.5g of dibutyltin dilaurate was added as a catalyst, and the mixture was stirred at 1000rpm for 5 hours at 75°C. Upon completion, the product was repeatedly washed with ethanol and centrifuged to eliminate excess silane and catalyst residues. Finally, the material was vacuum-dried at 60°C for 12 hours, producing 3-aminopropyl-triethoxysilane-functionalized graphene oxide (GO-GPTES).

Similarly, functionalization with KH-602 under the same experimental conditions resulted in the formation of GO-KH602.

2.3. Preparation of GO-GPTES/epoxy and GO-KH-602/epoxy coating

GO-GPTES powder (2 wt%) was initially dispersed in methyl isobutyl ketone to obtain a homogeneous suspension. Then, a predetermined amount of epoxy resin (EP) was introduced and mixed at high speed to form a uniform GO-GPTES/EP dispersion. A polyamide curing agent was then incorporated, and the mixture was stirred vigorously to ensure complete homogenization. Before application, the resulting formulation was filtered through a fine mesh to remove entrapped air bubbles.

Mild steel plates (CT3 grade, 70 x 100 x 1mm) were employed as substrates for coating deposition. The specimens were degreased with soap solution, dried, and mechanically polished with a wire brush followed by 400-grit abrasive paper to remove surface rust. The pretreated surfaces were sequentially rinsed with distilled water and absolute ethanol and dried.

The GO-GPTES/EP composite coating was applied to the pretreated steel substrates using a spray-coating technique. The dry film thickness was approximately 50 ± 5µm, as determined with an Ecometer thickness gauge. Similarly, the GO-KH-602 coating was fabricated following a procedure, namely GO-KH-602/EP.

An unmodified epoxy coating, prepared under identical conditions, served as the control sample.

2.4. Materials characterization and properties

Characterization

The morphological and elemental analyses were carried out using a field-emission scanning electron microscope (FE-SEM) with an energy-dispersive X-ray (EDX) spectrometer. Fourier transform infrared (FT-IR) spectra were obtained on a NEXUS 670 spectrometer within the wavenumber range of 400 - 4000cm^{-1} to investigate the chemical structure and surface functionalities of the prepared samples. The mechanical and physical properties of the material were characterized according to the applicable standards

Electrochemical measurements

Electrochemical impedance spectroscopy (EIS) was conducted on coated steel samples covered with the GO-GPTES/EP or GO-KH-602/EP film. A PVC tube containing 50mL of 3.5% NaCl solution was affixed to the surface of the coated specimen, exposing a working area of 16cm^2. The impedance spectra were recorded after immersion periods of 7, 30, and 60 days. Measurements were carried out in the frequency range from 100kHz to 10mHz using an automatic scan mode. A control sample coated with pure epoxy was also tested under the same conditions for comparison. All electrochemical measurements were performed using an AUTOLAB PGSTAT 302N workstation at the Institute of Chemistry and Materials.

3. RESULTS AND DISCUSSION

3.1. Characterization

FT-IR spectra

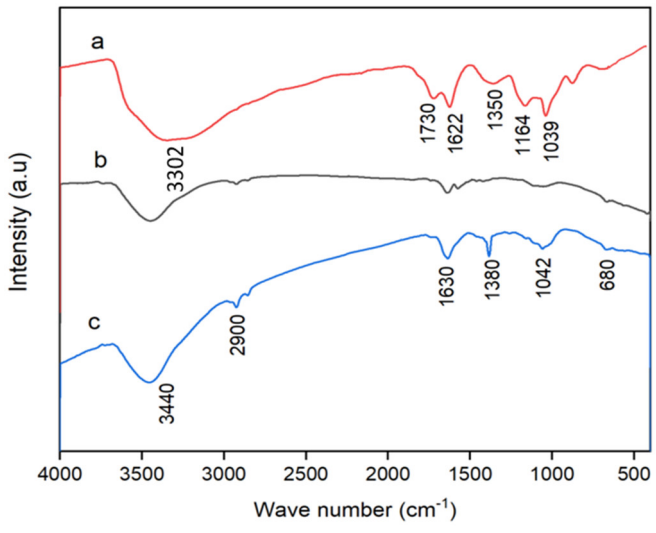


Figure 2. IR spectra of (a) GO, (b) GO-GPTES, (c) GO-KH-602 materials

The interactions between GO and the organic silane coupling agents GPTES and KH-602 were examined using Fourier transform infrared (FT-IR) spectroscopy. For comparison, pristine GO was also characterized under identical conditions. The corresponding FT-IR spectra are displayed in Figure 2.

In the FT-IR spectrum of GO (curve a), the broad peak at 3302cm^{-1} is attributed to the stretching vibration of hydroxyl (O–H) groups. The peak at 1730cm^{-1} corresponds to the C=O stretching of carbonyl functionalities, whereas the intense peak at 1622cm^{-1} is characteristic of the C=C skeletal vibration of the graphitic domains. In addition, the peaks at 1350cm^{-1} and 1164cm^{-1} are associated with C–O–H vibrations, while the peak at 1039cm^{-1} is assigned to C–O–C stretching, confirming the presence of oxygen-containing groups on the GO surface.

For GO functionalized with GPTES (curve b), distinct absorption features corresponding to both GO and GPTES can be clearly identified. The O–H stretching peak at 3303cm^{-1} exhibits a marked decrease in intensity, indicating limited consumption of hydroxyl groups through condensation with silane molecules. A new absorption peak in the 1100 - 1000cm^{-1} is assigned to Si–O–Si and Si–O–C stretching vibrations, confirming condensation reactions and forming a siloxane network on the GO surface. Furthermore, the reduced intensities of the epoxy and alkoxy peaks at 1164 and 1039cm^{-1}, respectively, suggest that the epoxy ring of GPTES was opened by hydroxyl groups on GO, leading to the formation of a stable Si–O–C covalent bond. These observations show that silanization with GPTES effectively grafted an organic siloxane framework onto the GO surface [6].

For GO modified with KH-602 (curve c), the peak is 3440cm^{-1}, corresponding to O–H and N–H stretching vibrations, while the peak at 2900cm^{-1} is associated with C–H stretching vibrations. The peak at 1630cm^{-1} is attributed to N–H bending vibrations, which may also reflect the formation of amide bonds or interactions between the –COOH groups of GO and the amino functionalities of KH-602. In addition, the peak at 1380cm^{-1} is assigned to CH_2/CH_3 vibrations, while the peak at 1042cm^{-1} corresponds to Si–O–Si and Si–O–C stretching vibrations. A low-frequency band at 680 cm^{-1} is related to Si–O and Si–C bending. Compared with pristine GO, the decreased intensity of the C=O peak at 1730cm^{-1} indicates that the carboxyl groups of GO reacted with the amino functionalities of KH-602 to generate a more stable interfacial structure [7].

FE-SEM images

FE-SEM images were employed to investigate the morphological evolution of GO before and after functionalization with silane coupling agents. The results are shown in Figure 3.

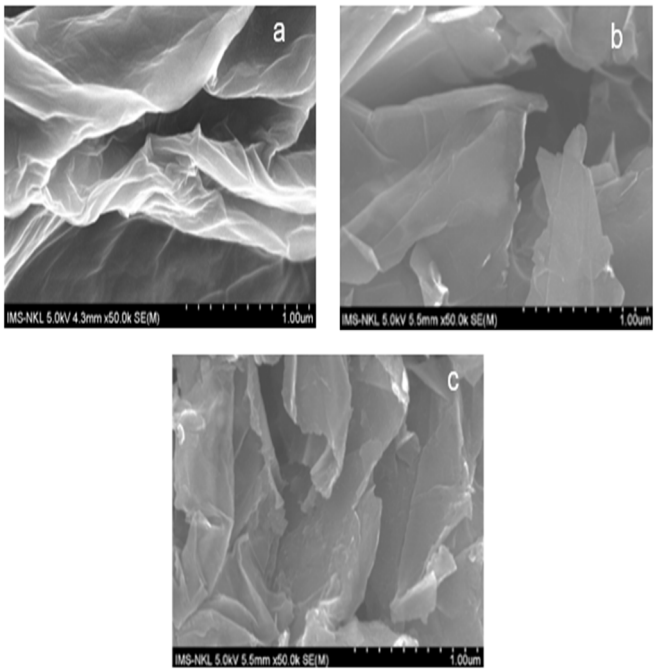


Figure 3. FE-SEM images of (a) GO, (b) GO-GPTES, (c) GO-KH-602 materials

The pristine GO sample (Figure 3a) exhibits thin, sheet-like structures composed of loosely stacked layers with abundant wrinkles and irregular edges, features that are characteristic of oxygenated functional groups disrupting the uniformity of the graphene framework. In contrast, GO functionalized with GPTES (Figure 3b) displays more aggregated and interconnected nanosheets with a denser and more uniform morphology, which can be attributed to covalent interactions between the epoxy/ethoxy groups of GPTES and the hydroxyl/epoxy functionalities on GO. Meanwhile, the GO modified with KH-602 (Figure 3c) exhibits a porous, three-dimensional network of irregularly distributed nanosheets with numerous pores. This morphology is most likely a result of chemical interactions, such as hydrogen bonding and covalent bonds, between the amino functionalities of KH-602 and the oxygen-containing groups of GO. These interactions disturb the regular stacking of GO layers and promote the formation of a porous structure.

These morphological features confirm the successful grafting of silane coupling agents onto GO, which is consistent with the FT-IR results.

EDX spectrocopy

Energy-dispersive X-ray (EDX) spectroscopy was utilized to analyze the elemental composition of GO before and after functionalization with organic silane coupling agents, with pristine GO serving as the reference. The elemental weight and atomic percentages were quantified based on five times selected measurement points for each sample, and the averaged results are presented in Table 1.

Table 1. Elemental composition of GO before and after organic silane modification

Element	GO		GO-GPTES		GO-KH-602	
	Weight (%)	Atom (%)	Weight (%)	Atom (%)	Weight (%)	Atom (%)
C	60.24	66.86	70.67	78.74	62.15	70.44
O	39.76	33.14	23.16	18.09	24.73	18.39
Si	-	-	6.17	3.17	7.91	6.42
N	-	-	-	2.22	5.21	4.75
Total	100	100	100	100	100	100

The results summarized in Table 1 demonstrate the difference in the elemental composition of GO before and after functionalization with organic silane coupling agents. In pristine GO, carbon constitutes 60.24 wt%, while oxygen accounts for a relatively high fraction (39.76 wt%), owing to abundant oxygen-containing functional groups on the surface. After functionalization with GPTES, the carbon content increases markedly to 70.67 wt%, with a significant reduction in oxygen (23.16 wt%) and the appearance of silicon (6.17 wt%). These results confirm the successful grafting of GPTES molecules onto the GO surface. Moreover, GO-KH-602 retains a higher oxygen content (24.73 wt%) compared with GO-GPTES; however, the significantly elevated silicon (7.91 wt%) and nitrogen (5.21 wt%) levels can be attributed to the presence of multiple silane and amine groups anchored on the surface.

3.2. Mechanical properties

The mechanical properties of epoxy coatings containing silane-modified GO were investigated at 1 and 2 wt% loadings. The results are presented in Table 2.

Flexural strength remained constant at 1mm for all coatings, indicating that GO incorporation did not affect film flexibility. Pencil hardness increased slightly from 4H at 1 wt% to 5H at 2 wt%, while adhesion reached the maximum rating (0 point) for GO-GPTES and GO-KH-602, showing improvement compared with pristine epoxy.

Impact resistance was the most sensitive parameter to GO addition. For GO-GPTES/EP coatings, the impact resistance increased from 125 to 140kg.cm as the filler content rose from 1 to 2 wt%, highlighting strong reinforcement. In contrast, GO-KH-602/EP exhibited lower impact values of 80 and 90kg.cm, reflecting limited toughening ability. Overall, GO-GPTES provided more effective mechanical enhancement than GO-KH-602, likely due to stronger interfacial interactions with the epoxy matrix.

Table 2. Mechanical properties of EP and silane-modified GO/EP coatings

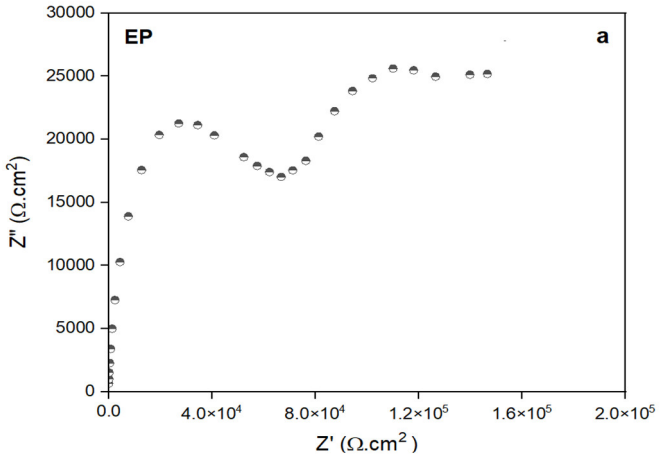
Coating type	Content, wt%	Flexural strength, mm	Hardness, H	Adhesion, grade	Impact resistance, kg.cm
EP	0	1	4	1	80
GO-GPTES	1	1	4	0	125
	2	1	5	0	140
GO-KH-602	1	1	4	0	80
	2	1	5	0	90

3.3. Anti-corrosion performance

Electrochemical impedance spectroscopy

Electrochemical impedance spectra were analyzed to assess the barrier properties of the coatings against electrolyte ingress. Higher impedance values indicate superior protection, with coating resistance above 10^9 Ω.cm^2 generally considered excellent, 10^6 - 10^9Ω.cm^2 adequate, and below 10^6Ω.cm^2 insufficient for reliable corrosion resistance [8].

The corrosion protection performance of silane-functionalized GO/epoxy (EP) coatings on CT3 steel was evaluated by electrochemical impedance spectroscopy in 3.5 wt% NaCl solution after immersion for 7 to 60 days. A pristine epoxy (EP) coating was also employed as a control for comparison.



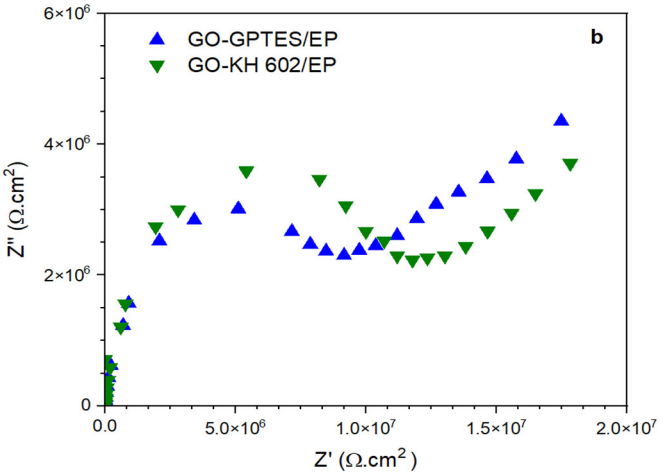


Figure 4. Nyquist impedance spectra of (a) pristine EP and (b) silane-functionalized GO/EP coatings after 7 days of immersion in 3.5 wt% NaCl solution

After 7 days of immersion, the Nyquist spectra of the coatings exhibited distinct differences in curve shape and impedance values. For the pristine EP coating (Figure 4a), two capacitive loops were clearly observed: the first loop at high frequencies corresponds to the barrier properties of the coating, whereas the second loop at low frequencies is associated with corrosion processes occurring at the coating-metal interface. The EP coating exhibited a resistance of 1.53×10^5Ω.cm^2 after 7 days, indicating a significant decrease in protective performance and the occurrence of steel corrosion.

In contrast, the Nyquist spectra of GO-GPTES/EP and GO-KH-602/EP coatings (Figure 4b) displayed a single capacitive loop at high frequencies and a diffusion-released line at low frequencies, which is characteristic of electrolyte ingress without corrosion of the metal. Both coatings exhibited similar resistance values of approximately 1×10^7Ω.cm^2, almost two orders of magnitude greater than that of the pristine EP coating. Thus, after 7 days of immersion, the impedance response of the silane-functionalized GO/EP coatings reflects contributions from both coating resistance and pore resistance within the coating structure.

To further demonstrate the long-term protective behavior, the Nyquist plots after 60 days of immersion in 3.5 wt% NaCl solution are presented in Figure 5.

The EP coating exhibited two distinct capacitive loops, evidencing corrosion processes at the metal-electrolyte interface. After 60 days of immersion, the Nyquist spectra of the GO-GPTES/EP and GO-KH-602/EP coatings still displayed a single capacitive loop without the development of a second loop (with coating resistance

reaching ~10^7Ω.cm^2), suggesting that electrolyte ingress through the coating, while significant metal corrosion had not yet initiated. Notably, GO-GPTES/EP showed slightly higher resistance than GO-KH-602/EP, possibly related to the denser silane–epoxy crosslinking induced by GPTES. The higher impedance of GO-GPTES/EP arises from epoxy–epoxy covalent crosslinking between GO and the epoxy matrix, leading to a more compact structure with fewer electrolyte diffusion pathways. In contrast, GO-KH-602/EP cures via an epoxy–amine mechanism, although improving adhesion, introduces polar groups (–NH, –OH) that may increase water affinity and slightly reduce long-term barrier performance. Nevertheless, both silane-functionalized GO/EP coatings exhibit markedly improved corrosion resistance compared with pristine epoxy, highlighting their effective shielding and protective capabilities.

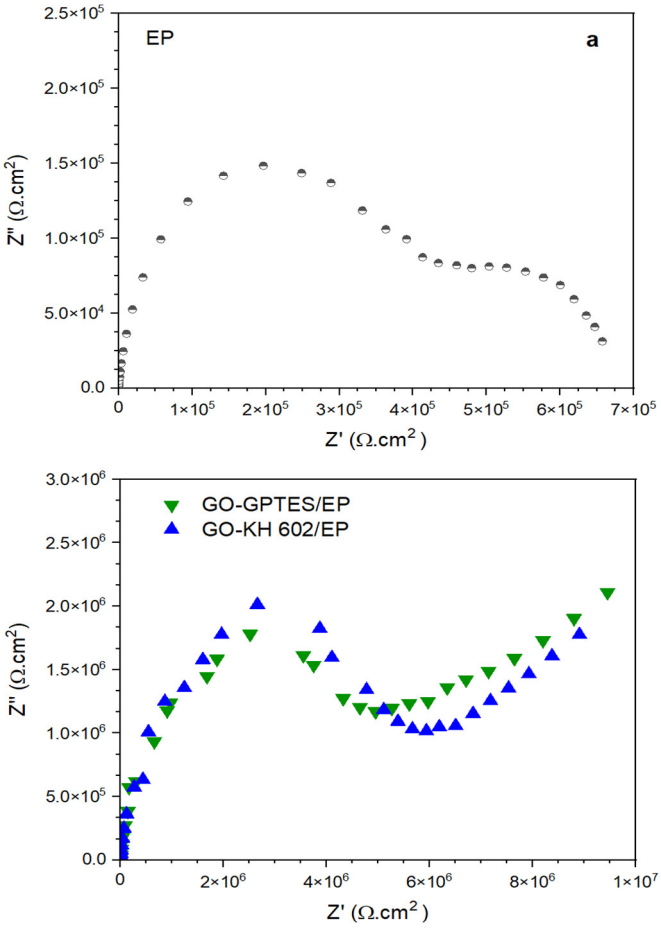


Figure 5. Nyquist impedance spectra of (a) pristine EP and (b) silane-functionalized GO/EP coatings after 60 days of immersion in 3.5 wt% NaCl solution

The impedance modulus at low frequency (Z_{10mHz}) is also a crucial parameter for assessing the corrosion durability of coatings, as it directly reflects the electrochemical processes at the metal surface. As summarized in Table 3, the Z_{10mHz} values of all coatings decreased progressively with immersion time. After 60 days, the silane-functionalized GO/EP coatings retained impedance values of 10^6 Ω.cm^2. In contrast, pristine epoxy dropped sharply to ~10^3Ω.cm^2, confirming the sustained corrosion protection afforded by the coatings.

Table 3. Low-frequency impedance modulus (10mHz) of materials

Time (day)	EP (Ω.cm²)	GO-GPTES/EP (Ω.cm²)	GO-KH-602/EP (Ω.cm²)
7	1.53×10^5	1.74×10^7	2.44×10^7
30	6.5×10^4	1.11×10^7	1.82×10^7
60	3.2×10^3	5.11×10^6	9.98×10^6

Electrochemical Evaluation by Tafel Polarization

The corrosion protection performance of silane-functionalized GO/EP coatings was further evaluated using Tafel polarization measurements. Figure 6 presents the polarization curves of functionalized GO/EP coatings deposited on CT3 steel substrates after 60 days of immersion in 3.5 wt% NaCl solution. A pristine EP coating was also examined as a reference for comparison. The electrochemical parameters calculated from these polarization curves, including the corrosion potential (E_{corr}) and corrosion current density (i_{corr}), are summarized in Table 4.

Compared with the pristine EP coating, the polarization curves of the silane-functionalized GO/EP coatings revealed a distinct shift toward lower current densities and more positive corrosion potentials in 3.5 wt% NaCl solution. After 60 days of immersion, the E_{corr} of the pristine EP/steel coating was measured at −463mV. In contrast, coatings containing GO-GPTES and GO-KH-602 exhibited significantly more positive values of −40mV and −39mV, respectively. Ahmadi et al. [9] reported that a positive shift in corrosion potential indicates anodic inhibition. This observation confirms that incorporating silane-functionalized GO into the epoxy matrix introduces a barrier effect and imparts an inhibitory action, leading to more positive corrosion potentials than pristine epoxy.

The corrosion current densities of the GO-GPTES/EP and GO-KH-602/EP coatings were determined to be 0.65 and 0.72nA.cm^{-2}, respectively, which are significantly lower than that of the pristine EP coating (10.26nA.cm^{-2}). This substantial reduction in i_{corr} is ascribed to the homogeneous dispersion of functionalized GO within the epoxy matrix, resulting in a dense and barrier layer that

effectively shields the steel substrate from the ingress of aggressive ions. Consequently, the increase in E_{corr}, coupled with the suppression of i_{corr}, highlights the significant reduction in the overall corrosion rate.

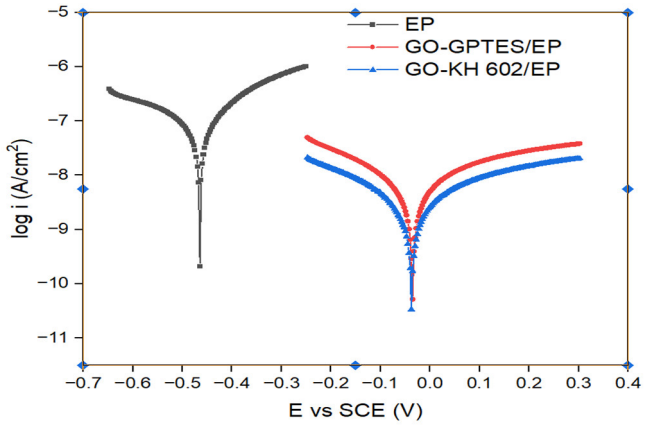


Figure 6. Tafel polarization curves of pristine EP and silane-functionalized GO/EP coatings on CT3 steel after 60 days of immersion in 3.5 wt% NaCl solution

Table 4. Electrochemical parameters derived from Tafel polarization

Material	E_{corr} (mV)	i_{corr} (nA/cm^2)	H %
Blank	- 463	10.26	-
GO-GPTES/EP	- 40	0.65	93.66
GO-KH-602/EP	- 39	0.72	92.98

The corrosion protection efficiency (H) of the coatings was calculated according to Equation:

$$H = \frac{i^o corr - i\ corr}{i^o\ corr}\ x\ 100\% \quad (1)$$

Where

i^o_{corr} is the corrosion current density of pristine EP in 3.5 wt% NaCl solution

i_{corr} is the corrosion current density of the silane-functionalized GO/EP coatings under identical conditions.

As shown in Table 3, the functionalized GO/EP coatings achieved protection efficiencies exceeding 90% after 60 days of immersion. These results demonstrate that the incorporation of silane-functionalized GO markedly enhances the long-term corrosion resistance of epoxy coatings, demonstrating its potential as an effective and environmentally sustainable strategy for metal protection.

4. CONCLUSION

In this study, silane-functionalized GO/epoxy coatings were successfully fabricated and evaluated for their corrosion protection performance on CT3 steel. Tafel polarization and impedance results confirmed that incorporating functionalized GO significantly reduced corrosion current density and shifted E_{corr} toward more positive values. GO-GPTES/EP and GO-KH-602/EP coatings achieved protection efficiencies above 90% under laboratory immersion conditions. The improved performance is attributed to the more uniform dispersion of silane-functionalized GO, which forms a dense barrier structure that slows electrolyte penetration. Among them, GO-GPTES/EP exhibited slightly higher resistance, suggesting that stronger crosslinking interactions were induced by epoxy-functional silane. These results highlight the potential of silane-functionalized GO as an effective nanofiller for enhancing the durability of epoxy anticorrosive coatings.

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