

Article

Preparation and Performance Study of Modified Graphene Oxide/Polyurethane Anti-Corrosion Coating

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Abstract

To address the corrosion and degradation of metallic materials in seawater, tidal, and similar environments, this study employs lysine ($C_6H_{14}N_2O_2$) to modify graphene oxide (GO) via a hydrothermal process. The modified graphene oxide (f-GO) and poly(l-lysine) (PL) composite was characterized structurally and functionally using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), thermogravimetric analysis (TGA), X-ray photoelectron spectroscopy (XPS), and scanning electron microscopy (SEM) to characterize its structure and properties. A composite coating was prepared using modified graphene oxide (f-GO) and polyurethane (PU), which underwent electrochemical testing, hardness testing, corrosion rate testing, adhesion testing, impact resistance testing, and salt spray corrosion resistance testing. Experimental results indicate that C-N stretching vibration peaks appeared at all reaction temperatures. At 85 °C, f-GO85 exhibited optimal modification with a layer spacing of 1.471 nm, 72% transmittance, and superior thermal stability, confirming successful lysine grafting onto the GO surface. Corrosion resistance testing of the composite coating revealed enhanced adhesion and impact resistance, reduced corrosion rate, decreased corrosion current density in polarization curves, positive shift in corrosion potential, and higher impedance values in impedance curves, indicating improved coating density and corrosion resistance. Salt spray tests demonstrated that incorporating lysine-modified graphene oxide significantly improved the anti-corrosion performance of polyurethane coatings. Optimal corrosion resistance was achieved when the modified graphene oxide content was 0.2 wt%.

Keywords: graphene oxide; lysine; polyurethane; anti-corrosion coating

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1. Introduction

In the field of marine engineering, metal materials are frequently exposed to seawater, salt spray, tidal environments, and other conditions during long-term service, accelerating corrosion and degradation [1–3]. This leads to severe performance deterioration of equipment, shortens its service life, and may even trigger safety hazards and significant economic losses. Statistics indicate that corrosion-related costs in China amount to approximately 2.1278 trillion yuan, accounting for about 3.34% of the country's GDP [4–6]. Applying anti-corrosion coatings to metal surfaces can delay corrosion while offering advantages such as ease of application, low cost, and strong adaptability. Polyurethane, a commonly used anti-corrosion coating, possesses characteristics similar to amide groups, exhibiting excellent abrasion resistance, mechanical properties, and adhesion. It is widely

applied in metal corrosion protection [7–9]. However, its poor corrosion resistance and water resistance due to hydrophilic groups can lead to diminished overall performance during long-term service in seawater environments. Consequently, enhancing the corrosion resistance of polyurethane has become a research hotspot in recent years.

Research has demonstrated that incorporating nanoparticles into polymers can enhance the overall performance of composite materials [10–13]. Graphene oxide, a two-dimensional nanomaterial characterized by abundant functional groups, high specific surface area, and superior mechanical strength [14–17], exhibits a shielding effect when filled into polyurethane, thereby improving the corrosion resistance of coatings [18,19]. However, graphene oxide contains abundant hydroxyl and carboxyl groups, and strong van der Waals forces between its layers make it prone to agglomeration [20–23]. To address this issue, researchers have modified graphene oxide to enhance its dispersion and stability. For instance, Ramezanzadeh et al. [24] modified graphene oxide with polyisocyanate (PI) and prepared PI-GO/PU nanocomposites, finding that the composite coating exhibited superior corrosion resistance and ion resistance compared to pure polyurethane. Li et al. [25] surface-modified graphene with titanate and incorporated it into a water-based polyurethane system to prepare a water-based polyurethane/graphene nanocomposite anti-corrosion coating. The study revealed that the composite coating exhibited optimal corrosion resistance at a graphene content of 0.4%. Zhang et al. [26] synthesized a functionalized silane coupling agent (2-(3,4-epoxycyclohexyl)ethyltriethoxysilane) to modify graphene oxide surfaces, addressing nanoparticle agglomeration issues. They enhanced the corrosion resistance of epoxy resin coatings by incorporating this functionalized graphene oxide. Results indicate that incorporating functionalized GO enhances the anti-corrosion performance of the epoxy composite coating, with the composite coating exhibiting optimal anti-corrosion performance at a functionalized GO mass fraction of 0.7 wt%. The above studies demonstrate that introducing modified GO can improve the corrosion resistance of coatings. However, most commonly used modifiers are toxic, and the modification process is relatively complex, making it difficult to meet green and environmentally friendly requirements.

L-lysine (L-Lys) is a naturally occurring amino acid with a diamine structure, offering advantages such as simple structure, abundant sources, non-toxicity, and biodegradability [27–30]. Simultaneously, its surface amino group can undergo amidation reactions with carboxyl groups and ring-opening reactions with epoxy groups on the GO surface, thereby enhancing GO dispersion and providing metal corrosion inhibition. Therefore, this study employs L-Lys-modified GO, combines it with PU to form a cured F/PU composite coating, and analyzes the coating's mechanical properties and anti-corrosion performance.

2. Materials and Methods

2.1. Materials and Instruments

Main raw materials: L-lysine (AR) (Shanghai McLean Biochemical Technology Co., Ltd., Shanghai, China); polyurethane prepolymer (AR) (Shandong Baiqian Chemical Co., Ltd., Jinan, China); curing agent MOCA (Shandong Bojiahe Chemical Technology Co., Ltd., Jinan, China); graphene oxide (AR) (Carbonene Technology Shenzhen Co., Ltd., Shenzhen, China); and ethyl acetate (AR) (Sinopharm Chemical Reagent Co., Ltd.).

Main experimental instruments: constant temperature water bath (HH-S model, Jiangsu Scientific Instrument Co., Ltd., Nanjing, China); adjustable high-speed homogenizer (FSH-2A model, Changzhou Wanfeng Instrument Manufacturing Co., Ltd., Changzhou, China); dual-frequency CNC ultrasonic cleaner (KQ-100VDB model, Kunshan Ultrasonic Instrument Co., Ltd., Nanjing, China); electric hot air drying oven (Model 101-0A, Tianjin Tester Instrument Co., Ltd., Tianjin, China); circulating water vacuum pump (Model SHZ-D,

Gongyi Yuhua Instrument Co., Ltd., Gongyi, China); electronic balance (Model YP4001, Shanghai Youke Instrument Co., Ltd., Shanghai, China); digital constant temperature magnetic stirrer (Model: Jindan Jingda Instrument Manufacturing Co., Ltd.); electrochemical workstation (Model: CHI660E, Gongyi Kerui Instrument Co., Ltd., Gongyi, China); scanning electron microscope (Model: TESCAN MIRA3, TESCAN Co., Ltd., China); Fourier transform infrared spectrometer (GD26-FTIR-650, Tianjin Zhongke Ruijie Technology Co., Ltd., Tianjin, China); X-ray photoelectron spectrometer (ESCALAB 250Xi, Thermo Fisher Scientific, USA); X-ray diffractometer (D8 Advance, Bruker, Germany); thermogravimetric analyzer (TGA55, Nanjing Nanda Wanhe Technology Co., Ltd., Nanjing, China); high-precision eddy current thickness gauge (Model TT260, Beijing Shidaizhifeng Instrument Co., Ltd., Beijing, China).

2.2. Experimental Content

2.2.1. Preparation of f-GO Powder

Weigh 0.5 g of graphene oxide powder and 0.1 g of lysine powder. Dissolve the weighed GO and lysine in 100 mL and 20 mL of ultrapure water, respectively, stir to dissolve, and then place in an ultrasonic cleaner for 40 min. Pour the thoroughly mixed lysine solution into the GO aqueous solution. Stir the mixture in a water bath at specific temperatures (65 °C, 75 °C, 85 °C, 95 °C, 105 °C) for 50 min. After the reaction is complete, remove the mixture and transfer it to an evaporating dish. Dry the mixture in a forced-air oven at 60 °C for 48 h. Remove the sample, grind it into powder in a mortar, collect, and set aside to obtain f-GO powder. Figure 1 illustrates the reaction mechanism of lysine-modified graphene oxide.

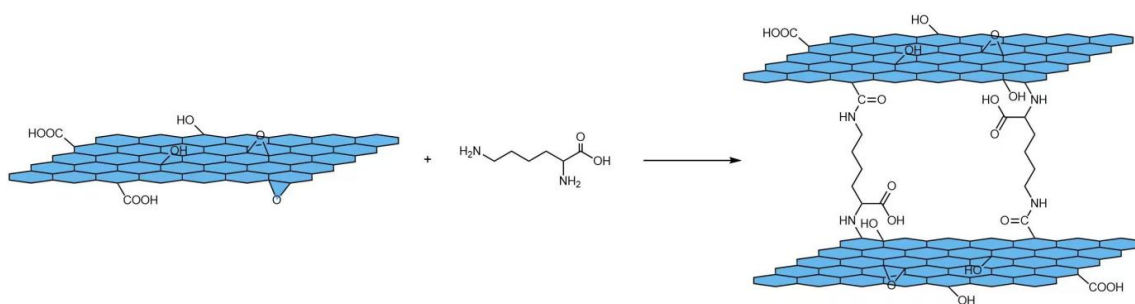


Figure 1. Mechanism diagram of lysine-modified graphene oxide reaction.

2.2.2. Preparation of F/PU Composite Coatings

The F/PU coating prepared in this study was obtained by mixing lysine-modified graphene oxide (f-GO) at concentrations of 0 wt%, 0.1 wt%, and 0.3 wt% with polyurethane prepolymer. The mixture was applied via dip-coating onto horizontally positioned Q235 steel plates and contained 0.2 wt% and 0.3 wt% lysine-modified graphene oxide (f-GO) with polyurethane prepolymer. This yielded four coating specimens with varying component contents: PU, F₁/PU, F₂/PU, and F₃/PU. The preparation process for F/PU coatings is as follows: Take a specific mass of polyurethane prepolymer and heat it to 80 °C for later use. Take an adequate amount of MOCA curing agent and heat it in another beaker at 115 °C until melted for later use. Weigh an equal mass of ethyl acetate dispersant into a beaker, add the corresponding mass of modified graphene, stir magnetically for ten minutes, then sonicate for two hours in an ultrasonic cleaner to ensure the modified graphene oxide is fully dispersed in the ethyl acetate. Pour the uniformly dispersed mixture into the preheated polyurethane monomer beaker and stir thoroughly. Maintain a polyurethane prepolymer to MOCA ratio of 100:18. Add the corresponding mass of MOCA to the mixture containing polyurethane monomer, ethyl acetate, and lysine-modified graphene oxide. Stir again until homogeneous and perform ultrasonic degassing. Using a pipette,

apply the mixture onto steel plates for coating curing. The procedure is as follows: Place the coated specimens in a constant-temperature, constant-humidity chamber. Initially cure at 25 °C and 50% relative humidity under ventilation for 48 h to ensure complete solvent evaporation and preliminary cross-linking, avoiding internal stress concentration or defects caused by rapid high-temperature curing. This is followed by post-curing: Maintain at 80 °C in a constant-temperature drying oven for 2 h to promote full cross-linking between the polyurethane prepolymer and MOCA curing agent. Finally, allow natural cooling to room temperature to complete the preparation of PU and F/PU coating specimens.

2.3. Testing and Analysis

2.3.1. X-Ray Diffraction Spectrum and Fourier Infrared Spectroscopy

Using a D8 Advance X-ray diffractometer with a Co target $K\alpha$ radiation wavelength $\lambda = 1.789 \text{ \AA}$, an accelerating voltage of 40 kV, a filament current of 15 mA, a scanning range (2θ) of 5° to 90°, and a scanning speed of 1° min^{-1} , diffraction patterns of GO and f-GO were obtained. Using a GD26-FTIR-650 Fourier transform infrared spectrometer, 0.2 mg of dried GO and f-GO were uniformly mixed with KBr at a specific mass ratio, ground, and pressed into pellets. Fourier transform infrared spectroscopy was performed with a wavenumber range of 4000 cm^{-1} to 400 cm^{-1} . Infrared spectra were obtained for GO and f-GO synthesized at different reaction temperatures.

2.3.2. Thermal Weight Loss Testing and Scanning Electron Microscopy

Testing was conducted using the TGA55 thermal loss tester by opening the pure nitrogen valve and adjusting the outlet pressure to 0.1 MPa. The morphology of graphene oxide and lysine-modified graphene oxide was characterized using a TESCAN MIRA3 scanning electron microscope.

2.3.3. X-Ray Photoelectron Spectroscopy and Electrochemical Testing

X-ray photoelectron spectroscopy (XPS) spectra of GO and f-GO were obtained using an ESCALAB 250Xi X-ray photoelectron spectrometer. A Mg target ($h\nu = 1253.6 \text{ eV}$) served as the X-ray source, operating at 10 kV and 20 mA. The energy resolution was set to 20 eV (narrow scan) and 100 eV (wide scan). A charge neutralizer was employed during testing to eliminate surface charging effects on the sample. The C 1s peak (284.8 eV) served as the calibration reference for binding energy. Polarization curves and electrochemical impedance spectra were measured using a CHI660E electrochemical workstation for Q235 steel plates coated with 0%, 0.1%, 0.2%, and 0.3% modified graphene oxide polyurethane. Samples were immersed for 1 day and 10 days in an undegassed 3.5 wt% NaCl solution at a test temperature of 25 °C.

2.3.4. Adhesion Testing and Impact Resistance Testing

Adhesion testing of coated specimens was conducted using an adhesion pull-off tester (PS 2658). Dry adhesion was measured on specimens not immersed in 3.5 wt% sodium chloride solution, while wet adhesion was measured on specimens immersed in 3.5 wt% sodium chloride solution for 36 h. The adhesion loss rate was calculated according to Formula (1). The equipment technical parameters used were 1 MPa/s with a 20 mm diameter puller. The impact resistance of the coating was tested according to GB/T 1732-2020, the method for determining paint film impact resistance. The spacing between test panels—specifically, the distance between the edge of the impact point and the edge of the

test panel—was no less than 12 mm. The distance between the edges of adjacent impact points was also no less than 12 mm.

$$\text{Loss of wet and dry adhesion} = \left(\frac{\text{Dry adhesion} - \text{Wet adhesion}}{\text{Dry adhesion}} \right) \times 100\% \quad (1)$$

2.3.5. Hardness Testing and Corrosion Rate Testing

Scratch testing of the coating surface was conducted using a pencil hardness tester or an equivalent hardness-testing instrument. Following the method specified in GB/T6739-2006 (Determination of Hardness of Paint Films by Pencil Method for Paints and Varnishes), we selected 13 hardness gradients ranging from 6B to 6H. The pencil was sharpened to a flat tip while maintaining a smooth point. Under a 750 g load, the pencil was inserted at a 45° angle into the testing apparatus and slid at a uniform speed. Each hardness grade was tested three times consecutively, with progressively increasing hardness levels. If no penetrating scratch appears after two or more passes, the coating is deemed to meet that pencil hardness grade. Corrosion rate was measured using the immersion weight-loss method. A coating specimen with a known exposed area was cleaned with anhydrous ethanol, vacuum-dried at 60 °C to constant weight, and weighed to obtain the initial mass m_0 (non-test surface sealed). It was then fully immersed in a corrosion medium maintained at 25 °C for 10 days. After removal, corrosion products were removed, the specimen was cleaned and dried, and the final mass m_t was weighed. The corrosion rate was calculated using Formula (2).

$$CR_w = \frac{8.76 \times 10^4 \times (m_0 - m_t)}{\rho \times A \times t} \quad (2)$$

In the formula:

- Corrosion rate, mm/y;
- Sample mass before and after corrosion, g;
- Base metal density, g/cm³;
- Exposed area of the specimen, cm²;
- Corrosion time, h;
- Unit conversion factor.

2.3.6. Dry Film Thickness Measurement

Using a high-precision eddy current thickness gauge (Model: TT260, measuring range 0–1000 µm, accuracy ±1 µm), in accordance with GB/T 13452.2-2008 “Paints and Varnishes—Determination of Film Thickness,” we randomly selected 5 test points on the surface of each coating specimen that were free of scratches and defects. After excluding outliers, we calculated the mean and standard deviation. For each coating formulation, we prepared 3 parallel samples ($n = 3$). The final dry film thickness is the average value of the 3 samples ± standard deviation.

3. Results and Discussion

3.1. Characterization of GO and f-GO Structures

3.1.1. XRD Characterization

Figure 2 shows the XRD patterns of GO and L-lysine-modified GO (f-GO) at different temperatures, used to analyze their microstructure and interlayer spacing. GO exhibits a characteristic (001) peak at $2\theta = 11.79^\circ$. After modification, the diffraction peaks of f-GO shift to 9.2° , 8.4° , 6° , 9.6° , and 9° , indicating that lysine introduction increases the interlayer spacing. Calculations based on the Bragg equation yielded interlayer spacings of 0.749, 0.96, 1.052, 1.471, 0.92, and 0.982 nm, respectively, with the maximum interlayer

spacing (1.471 nm) observed at 85 °C modification. Lysine reacts with carboxyl and epoxy groups on the GO surface, introducing numerous molecular chains and functional groups that effectively increase the interlayer spacing. At temperatures below 85 °C, insufficient reaction kinetics result in low lysine grafting efficiency and minimal interlayer spacing expansion. At temperatures exceeding 85 °C, excessive heat causes partial decomposition or over-reaction of lysine molecules, leading to a decrease in interlayer spacing. Therefore, the maximum interlayer spacing observed at 85 °C directly reflects the optimal progression of the modification reaction and serves as the key basis for determining this temperature as the optimal modification condition.

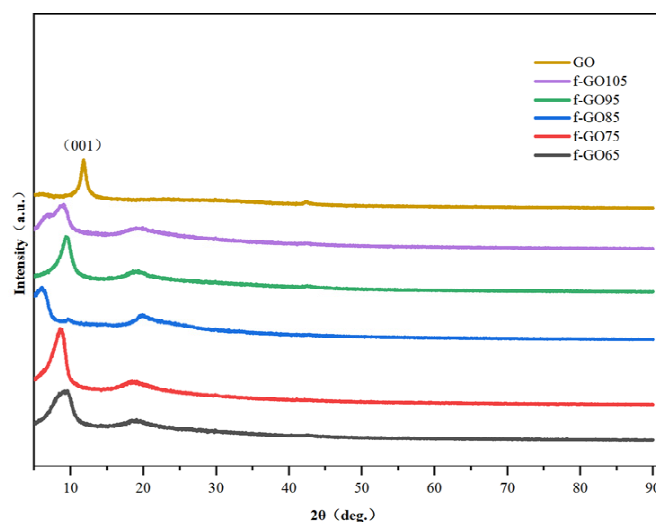


Figure 2. XRD pattern.

3.1.2. FT-IR Characterization

The molecular structures of graphene oxide and its derivatives were characterized using an infrared spectrometer. Figure 3a shows the infrared spectra of GO and modified graphene oxide at 65 °C, 75 °C, 85 °C, 95 °C, and 105 °C. The infrared spectrum of GO reveals functional groups including O-H, C-H, carboxyl C=O, C=C, and C-O. After modification, the amino group of lysine reacts with the carboxyl group in GO to form amide bonds, generating characteristic absorption peaks at 1620 cm⁻¹ (amide I), 1514 cm⁻¹ (amide II), and 1362 cm⁻¹ (amide III). Concurrently, the intensity of the carboxyl C=O peak diminishes, confirming successful amidation. Absorption peaks at 3585 cm⁻¹ (O-H and N-H) and 2943 cm⁻¹ (C-H stretching vibration) further confirm the successful grafting of lysine onto GO. After normalization, the transmittances of f-GO at different temperatures were 88%, 78%, 72%, 91%, and 81%, respectively, as shown in Figure 3b. The sample at 85 °C exhibited the lowest transmittance and the strongest functional group peaks, indicating optimal lysine modification efficiency at this temperature.

3.1.3. Thermogravimetric Analysis

Figure 4 shows the thermogravimetric analysis of GO and modified graphene oxide at 65 °C, 75 °C, 85 °C, 95 °C, and 105 °C. The yellow curve represents unmodified GO, which exhibits severe weight loss around 200 °C and drops to less than 10% of its initial mass by 300 °C, indicating poor thermal stability. In contrast, all f-GO samples exhibit thermal decomposition onset temperatures higher than GO, indicating enhanced thermal stability through modification. f-GO65 and f-GO75 still show significant weight loss between 200 and 300 °C, suggesting incomplete modification at lower temperatures. Thermal stability increases with rising modification temperature, shifting the curve to the right. At 700 °C, the residual mass comparison is f-GO85 > f-GO105 > f-GO95 > f-GO75 > f-GO65 > GO,

with f-GO85 exhibiting the highest residual mass. At 700 °C, the residual mass comparison was f-GO85 > f-GO105 > f-GO95 > f-GO75 > f-GO65 > GO. f-GO85 exhibited the highest residual mass and a smoother thermal decomposition curve, indicating that 85 °C is the optimal modification temperature. At this temperature, lysine achieves the most complete bonding with GO, effectively reducing the formation of unstable functional groups and significantly enhancing GO's thermal stability.

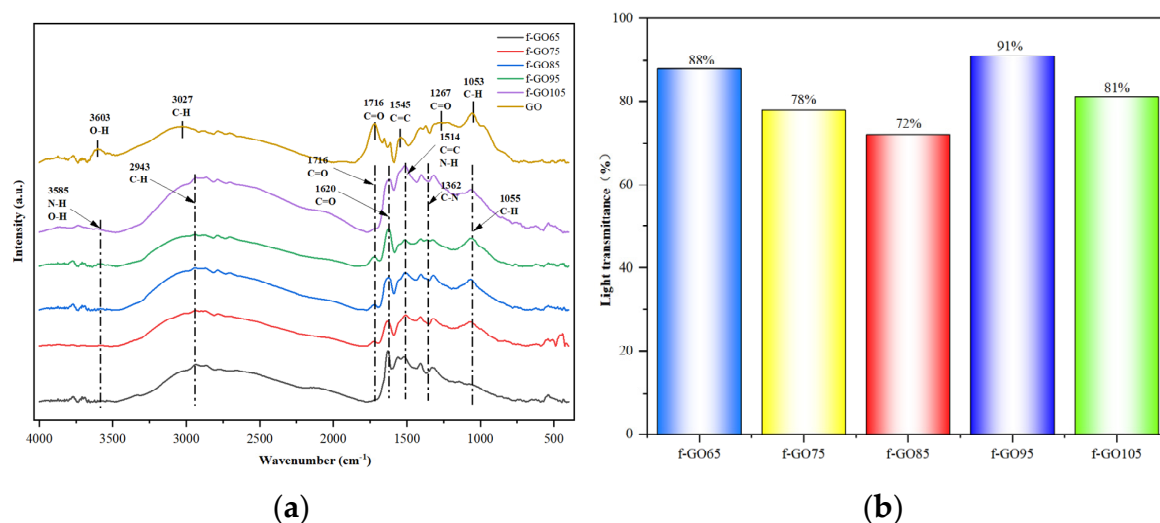


Figure 3. FT-IR spectrum and transmittance: (a) FT-IR spectrum; (b) transmittance.

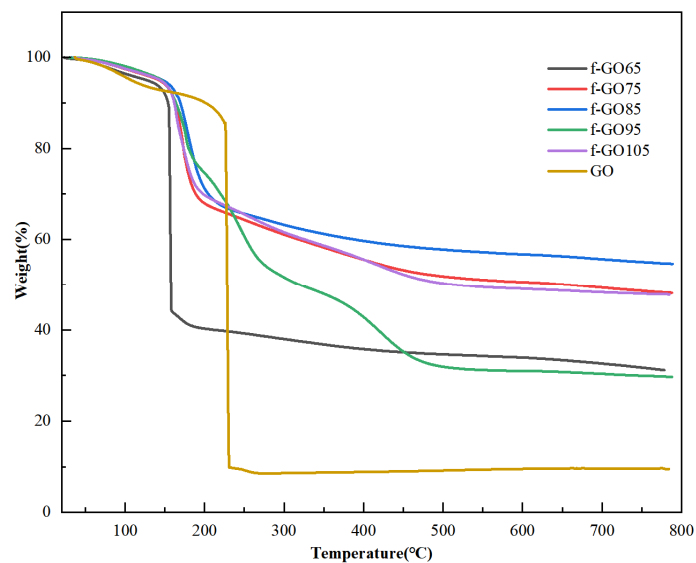


Figure 4. TGA curve.

3.1.4. SEM Analysis

Figure 5 shows scanning electron micrographs of GO and modified graphene oxide (f-GO85) synthesized at the optimal reaction temperature. Figure 5a,c presents low-magnification SEM images of GO and f-GO85, respectively. As observed in Figure 5a,b, the surfaces exhibit distinct wrinkles and layered structures; GO flakes are relatively smooth and flexible, exhibiting good flattening and layered structure. Magnified observation of the edges clearly reveals GO's single-layer sheet structure, with flakes stacked upon each other. This stacking arises from van der Waals forces between GO particles, causing aggregation and accumulation. Figure 5b,d presents high-magnification SEM images of GO and f-GO85, respectively. Figure 5c,d reveals slight alterations in the graphene layer morphology,

with localized fractures, deformations, or stacked adhesion phenomena. These particles are considered lysine molecules or reaction products formed between lysine and the GO surface. The interlayer spacing slightly increases after modification, enhancing material dispersibility and functional group exposure. Lysine modification does not disrupt the layered structure of graphene oxide. Local magnification reveals folded edges and irregular morphologies formed by ultrasonic exfoliation. The rough surface structure indicates successful grafting of lysine onto the GO surface.

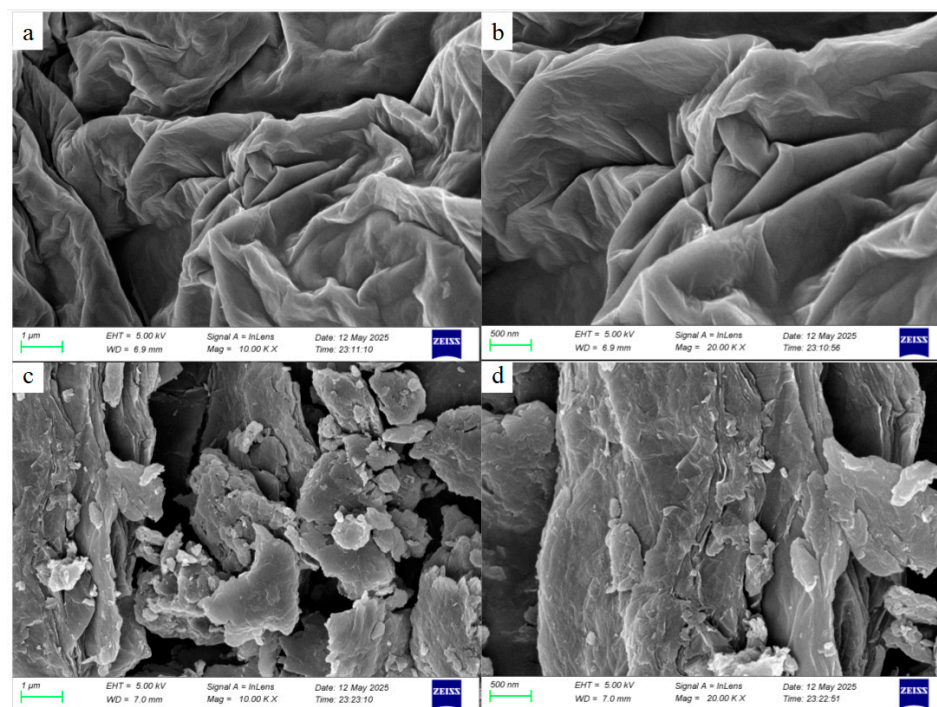


Figure 5. SEM morphology of GO and modified graphene oxide at optimal reaction temperature: (a) SEM of GO; (b) local high-magnification morphology of GO; (c) SEM of f-GO85; and (d) local high-magnification morphology of f-GO85.

3.1.5. XPS Characterization

XPS analysis shows that the full spectrum of GO in Figure 6a exhibits characteristic peaks of C1s (~286.74 eV) and O1s (~530.71 eV), while the f-GO85 in Figure 6b additionally shows an N1s peak (~400.8 eV), indicating the introduction of nitrogen-containing groups. The amine group in lysine reacts with carboxyl groups on the GO surface to form amide bonds, while simultaneously undergoing substitution or ring-opening reactions with epoxy or hydroxyl groups. This leads to enhanced N1s and weakened O1s signals. Table 1 data show that oxygen content in f-GO85 decreased from 32.8% to 20.1%, while nitrogen content increased to 9.7%, confirming successful lysine grafting onto the GO surface, introducing nitrogen and partially replacing oxygen functional groups. The high-resolution C1s spectrum of GO in Figure 6c primarily exhibits C-C (284.8 eV) and C-O (286.8 eV) peaks. Although the modified f-GO85 (Figure 6d) retains similar oxygen-containing groups, the C-O peak intensity is significantly weakened, indicating reduced hydroxyl and epoxy groups. This suggests that lysine partially reduces and structurally modifies GO. Simultaneously, no distinct C=O peak was observed at 288.2 eV, indicating that some carboxyl groups underwent amidation with lysine, resulting in reduced carboxyl content.

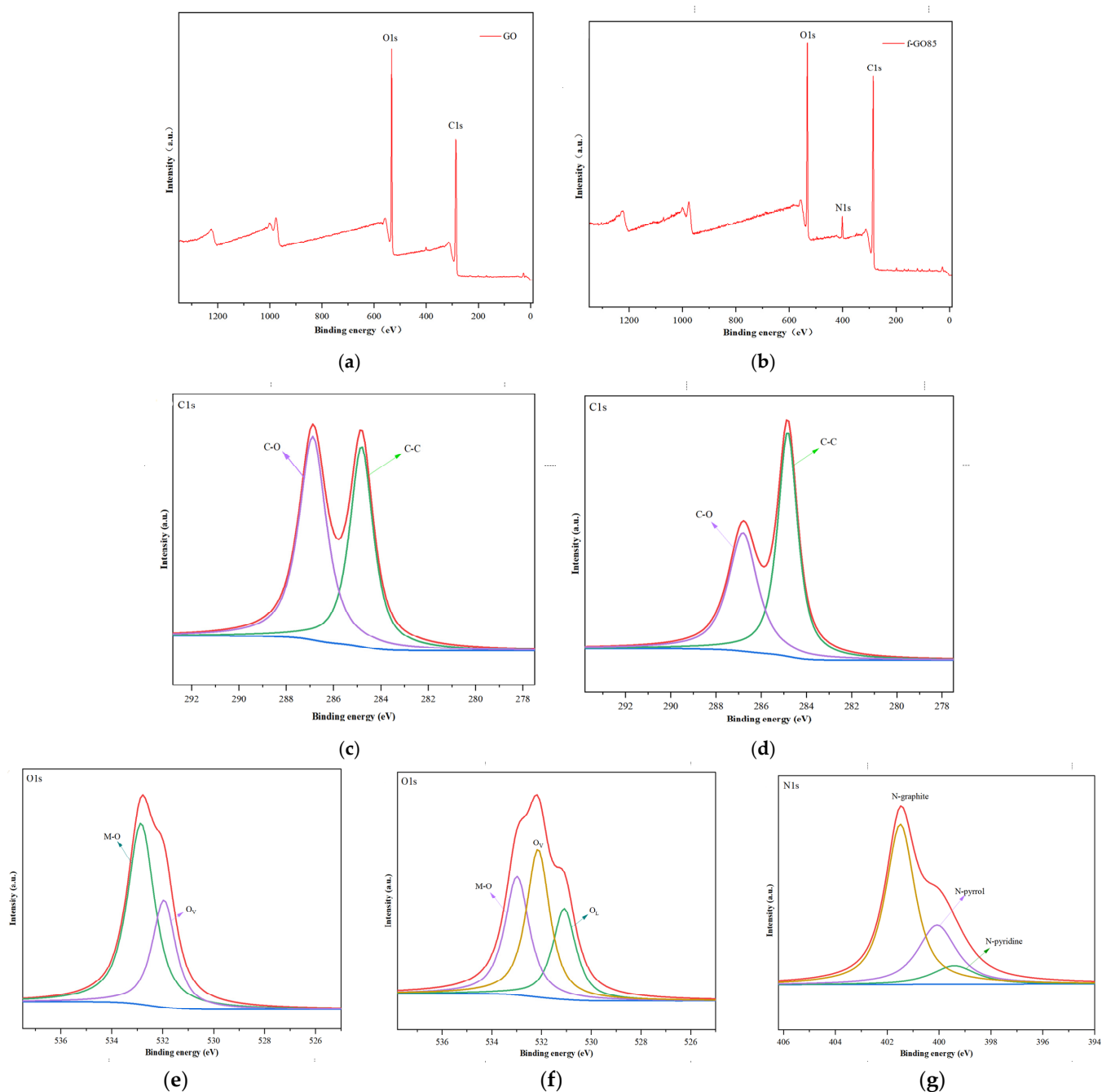


Figure 6. XPS spectra of GO and f-GO85: (a) XPS spectrum of GO; (b) XPS spectrum of f-GO85; (c) high-resolution C1s spectrum of GO; (d) high-resolution C1s spectrum of f-GO85; (e) high-resolution O1s spectrum of GO; (f) high-resolution O1s spectrum of f-GO85; and (g) high-resolution N1s spectrum of f-GO85.

Table 1. XPS carbon, oxygen, and nitrogen atomic percentages.

Sample	C (at%)	O (at%)	N (at%)
GO	67.2	32.8	0
f-GO85	70.2	20.1	9.7

Figure 6e,f shows the O1s scanning spectra for GO and f-GO85, respectively. The peaks at 532.81 eV and 531.9 eV in GO correspond to C-O and C=O bonds, while f-GO85 exhibits peaks at 531.8 eV, 533.0 eV, and 530.4 eV. The new peak at 530.4 eV is characteristic

of amide bonds (-CO-NH-), indicating that the amino group of lysine has formed covalent amide bonds with the carboxyl groups of GO.

Figure 6g shows the N1s spectrum of f-GO85, revealing pyridine nitrogen at 398.4 eV, pyrrole nitrogen at 400.1 eV, and graphite nitrogen at 401.2 eV. This confirms that lysine not only successfully introduced nitrogen elements but also doped into the GO carbon framework, forming stable nitrogen-functionalized structures.

3.2. F/PU Performance Analysis of Composite Coatings

3.2.1. Adhesion Analysis

Adhesion performance tests were conducted on pure polyurethane (PU) coatings and composite coatings (F₁/PU, F₂/PU, F₃/PU) containing lysine-modified graphene oxide (f-GO) at 0.1 wt%, 0.2 wt%, and 0.3 wt% additions. Figure 7 shows the comparison results for dry adhesion (unsoaked), wet adhesion (soaked in 3.5 wt% sodium chloride solution for 36 h), and adhesion loss rate. As shown in Figure 7a, the dry adhesion strengths of PU, F₁/PU, F₂/PU, and F₃/PU were 2.16 MPa, 1.66 MPa, 1.87 MPa, and 1.82 MPa, respectively. The initial adhesion strengths of the coatings showed minor differences, with the pure PU coating exhibiting the highest dry adhesion. After 36 h of salt solution immersion, the wet adhesion of the aforementioned coatings was 1.21 MPa, 1.23 MPa, 1.46 MPa, and 1.37 MPa, respectively. Evidently, the PU coating exhibited the lowest wet adhesion, while the F₂/PU coating demonstrated the highest wet adhesion (1.46 MPa). Furthermore, the pure PU coating experienced the greatest decrease in adhesion from dry to wet conditions. All modified composite coatings showed higher wet adhesion than the pure PU coating, indicating that the modified fillers effectively enhance interfacial adhesion between the coating and substrate in corrosive environments. The adhesion loss rate results are shown in Figure 7b: the pure PU coating exhibited a high loss rate of 44%, while the modified coating F₂/PU had the lowest loss rate (21.9%). This result further confirms that lysine-modified graphene oxide can significantly suppress the destructive effects of corrosive media on the coating-substrate interface.

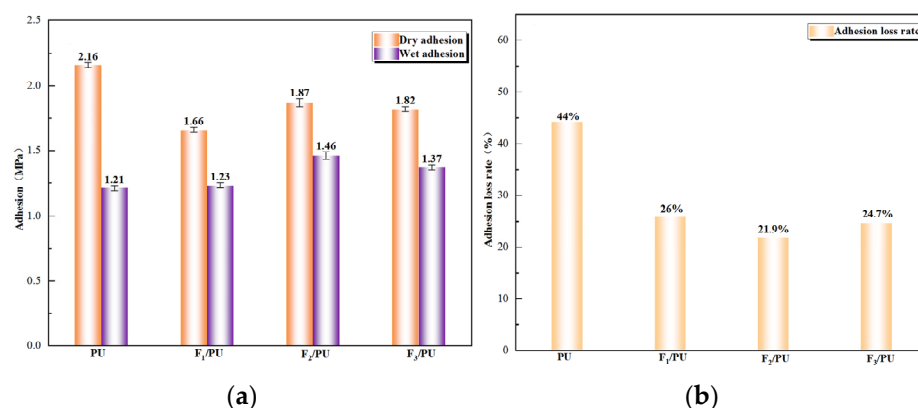


Figure 7. Comparison of adhesion performance metrics for PU and F/PU coatings: (a) dry and wet adhesion; (b) adhesion loss rate.

3.2.2. Corrosion Rate

Figure 8 shows the corrosion rate trends of coatings with different graphene oxide contents in H₂O, NaOH, and NaCl media. Results indicate that as the f-GO content increases, the coating's corrosion rate first decreases and then increases, with the lowest corrosion rate observed at 0.2 wt% f-GO, indicating the best corrosion resistance. Compared to pure PU coatings, F₂/PU coatings exhibited significantly reduced corrosion rates in all three media. This indicates that an appropriate amount of f-GO enhances coating density and delays the penetration of corrosive media. When the content further increased

to 0.3 wt%, the corrosion rate slightly rebounded. This may be attributed to localized agglomeration and the formation of defect pathways caused by excessive f-GO, thereby weakening the protective effect. Comprehensive analysis indicates that 0.2 wt% is the optimal addition level, significantly improving the coating's corrosion resistance.

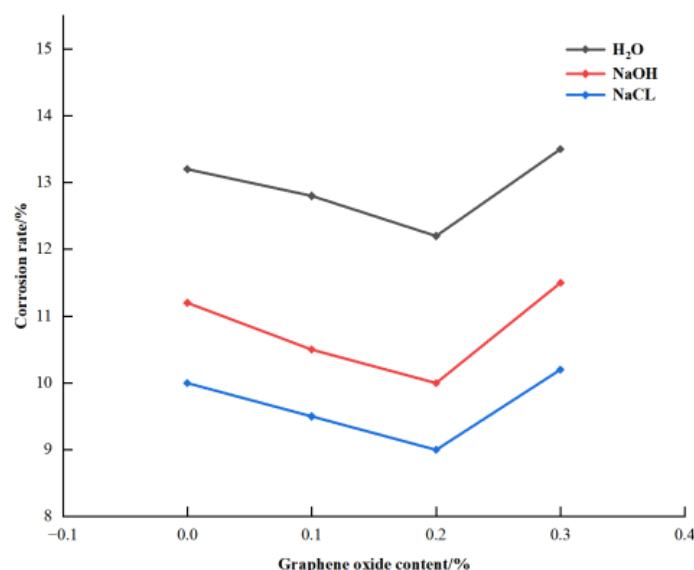


Figure 8. Corrosion rates under different corrosive media.

3.2.3. Impact Resistance Analysis

Impact resistance is one of the key factors determining the service life of coatings; generally, the better the performance, the longer the coating lasts. The impact resistance test results are shown in Figure 9. The results indicate that the pure polyurethane (PU) coating exhibits the poorest impact resistance, while the introduction of lysine-modified graphene oxide (f-GO) significantly enhances the coating's impact resistance. When the f-GO content was 0.1 wt%, the forward impact strength of the F₁/PU coating increased from 81 kg·cm to 89 kg·cm, and the reverse impact strength rose from 27 kg·cm to 34 kg·cm. When the f-GO content increased to 0.2 wt%, the forward impact strength of the F₂/PU coating reached a peak of 96 kg·cm, while the reverse impact strength substantially increased to 46 kg·cm. Further increasing the f-GO content to 0.3 wt% caused a slight decrease in forward impact strength, but the reverse impact strength continued to rise to 48 kg·cm. These results indicate that lysine-modified graphene oxide enhances the toughness of polyurethane coatings by strengthening interfacial bonding and dispersing stress. The sustained improvement in reverse impact strength directly reflects the modification component's optimization of the coating's resistance to deformation failure, while the slight fluctuation in forward impact strength may be related to the dispersion state of f-GO at high loading levels. This study provides experimental evidence for controlling the dosage of lysine-modified graphene oxide in polyurethane coatings and elucidates its impact resistance enhancement mechanism.

3.2.4. Hardness Analysis

Scratch tests were conducted on the coated PU, F₁/PU, F₂/PU, and F₃/PU specimens using a pencil hardness tester or equivalent hardness tester in accordance with the method specified in GB/T 6739-2006 (Paints and Varnishes—Pencil Test for Determining the Hardness of Paint Films). The test results are shown in Table 2.

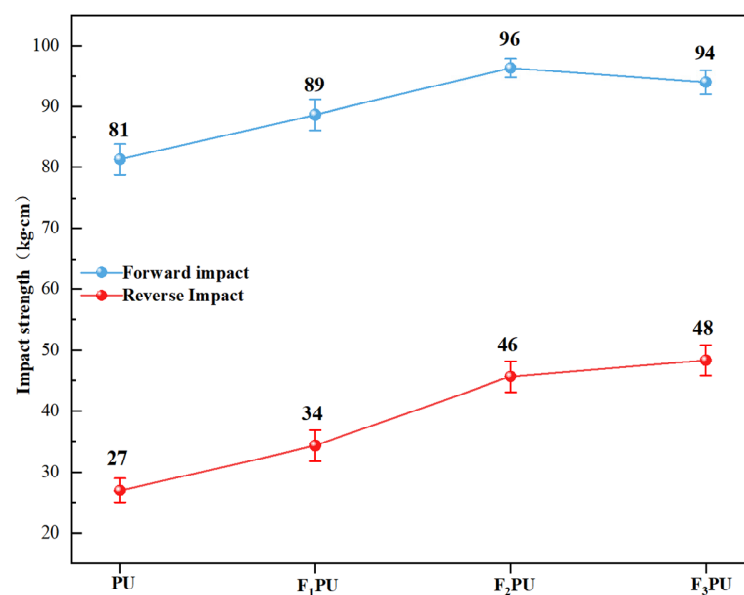


Figure 9. Comparison of forward and reverse impact resistance for PU and F/PU coatings.

Table 2. Hardness testing of PU and F/PU coatings.

Coating Type	PU	F ₁ /PU	F ₂ /PU	F ₃ /PU
Pencil hardness	4H	5H	6H	6H

3.2.5. Dry Film Thickness Analysis

The dry film thickness of the coated PU, F₁/PU, F₂/PU, and F₃/PU specimens was measured according to the method specified in GB/T 13452.2-2008 (“Paints and Varnishes—Determination of Film Thickness”). As shown in Table 3, all coatings maintained dry film thicknesses within the 65–71 µm range, with minimal variation between groups. This excludes thickness as a confounding factor in the mechanical and corrosion resistance test results. With increasing f-GO content, the dry film thickness showed a slight increase. This is attributed to the uniform dispersion of f-GO flakes within the polyurethane matrix, which slightly increased the coating’s bulk density without causing significant thickness anomalies. This outcome aligns with the practical application requirements for anti-corrosion coatings.

Table 3. Test of dry film thickness of PU and F/PU coatings.

Coating Type	PU	F ₁ /PU	F ₂ /PU	F ₃ /PU
Dry film thickness (µm)	65.2	68.5	70.1	69.3

3.3. Electrochemical Performance Analysis of Coatings

3.3.1. Electrochemical Impedance Spectroscopy (EIS) Analysis

Figure 10 shows the electrochemical impedance curves of polyurethane composite coatings modified with graphene oxide (f-GO) at different lysine concentrations after immersion in a 3.5 wt% NaCl solution for 10 days. The figure reveals that coatings incorporating lysine-modified graphene oxide exhibit a significantly larger “semicircle diameter” in the Nyquist plot compared to unmodified coatings, indicating that lysine-modified graphene oxide substantially enhances coating resistance. Among the various modified graphene oxide concentrations, the F₂/PU coating exhibits the largest semicircle diameter, signifying the highest resistance value and optimal protective performance at this concentration. As the f-GO content increased to 0.3 wt%, the impedance slightly decreased,

possibly due to excessive filler agglomeration and reduced dispersion. In the Bode plot, a higher impedance modulus $|Z|$ indicates stronger current-blocking capability of the coating and superior corrosion resistance. Simultaneously, Figure 10b shows that $|Z|$ for coatings containing f-GO consistently exceeds that of the f-GO-free control across the entire frequency range. This confirms that f-GO addition effectively enhances the overall impedance of the coating. Among the tested concentrations, the coating with 0.2 wt% modified graphene oxide exhibits the highest impedance modulus $|Z|$ across the entire frequency spectrum. Although the 0.1 wt% modified graphene oxide coating shows some improvement compared to the unmodified coating, its overall protective performance is inferior to that of the 0.2 wt% modified graphene oxide coating. The impedance modulus $|Z|$ of the 0.3 wt% modified graphene oxide coating is below 0.2%, consistent with the trend observed in the Nyquist plot. This performance decline is also attributed to the agglomeration of modified graphene oxide. Overall, the incorporation of lysine-modified graphene oxide significantly enhances the corrosion resistance of polyurethane composite coatings, with optimal anti-corrosion performance achieved at a 0.2 wt% addition level.

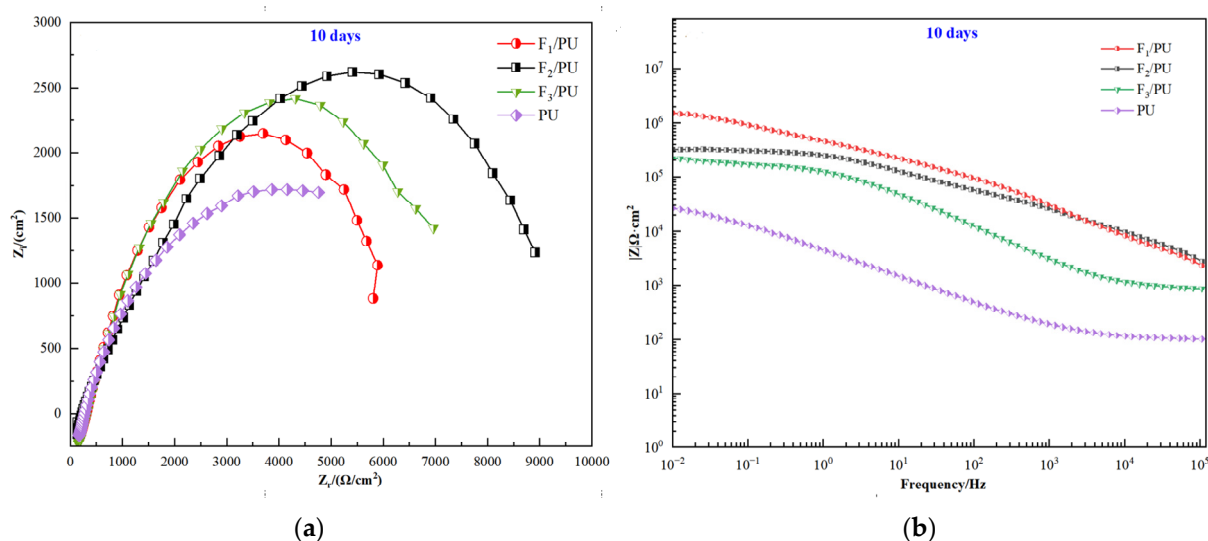


Figure 10. Electrochemical impedance test results of different coatings after 10 days of immersion in a 3.5 wt% NaCl solution: (a) Nyquist plot; (b) Bode plot.

3.3.2. Tafel Polarization Analysis

Figure 11a,b shows the polarization curves after immersion in a 3.5 wt% NaCl solution for 1 day and 10 days, respectively. Table 4 presents the E_{corr} , i_{corr} , and Tafel slope results for polyurethane coatings with varying amounts of modified graphene oxide (f-GO) in a 3.5% NaCl solution. The polarization curves after 1-day and 10-day immersion indicate that all f-GO-containing PU coatings outperform the pure PU coating in corrosion current density. The pure PU sample exhibits the highest polarization current density (maximum $\log |I|$), whereas increasing f-GO content results in lower current densities in the low-frequency/corrosion region of the polarization curve, demonstrating superior barrier/corrosion inhibition capability. Table 4 data reveal that F₂/PU coatings exhibited the most positive corrosion potential (E_{corr}) among all groups after both 1-day and 10-day immersion (1d: -0.38 ± 0.02 V, 10d: -0.32 ± 0.01 V) and the lowest corrosion current density (i_{corr}) (1d: $(8.54 \pm 0.36) \times 10^{-8}$ A/cm², 10d: $(1.23 \pm 0.15) \times 10^{-8}$ A/cm²), while the pure PU coating exhibited the most negative E_{corr} and highest i_{corr} . This clearly demonstrates the optimal improvement in corrosion resistance achieved with 0.2 wt% f-GO. Simultaneously, the Tafel slope fluctuations across all groups were minimal, indicating that f-GO did not alter the corrosion reaction kinetics but optimized anti-corrosion performance

solely through physical barrier effects. Overall, the F₂/PU and F₃/PU coatings significantly reduced polarization currents under both immersion durations. The incorporation of lysine-modified graphene oxide markedly enhanced the corrosion resistance of PU coatings. Furthermore, the small standard deviation in repeated experiments validated the data's reproducibility and reliability. These quantitative results fully align with the optimal performance of the F₂/PU coating observed in polarization curves and EIS spectra. Compared to pure PU, the F₂/PU coating exhibited lower polarization current density and more stable polarization curves, indicating its superior effectiveness in delaying ion/moisture penetration and inhibiting electrochemical corrosion.

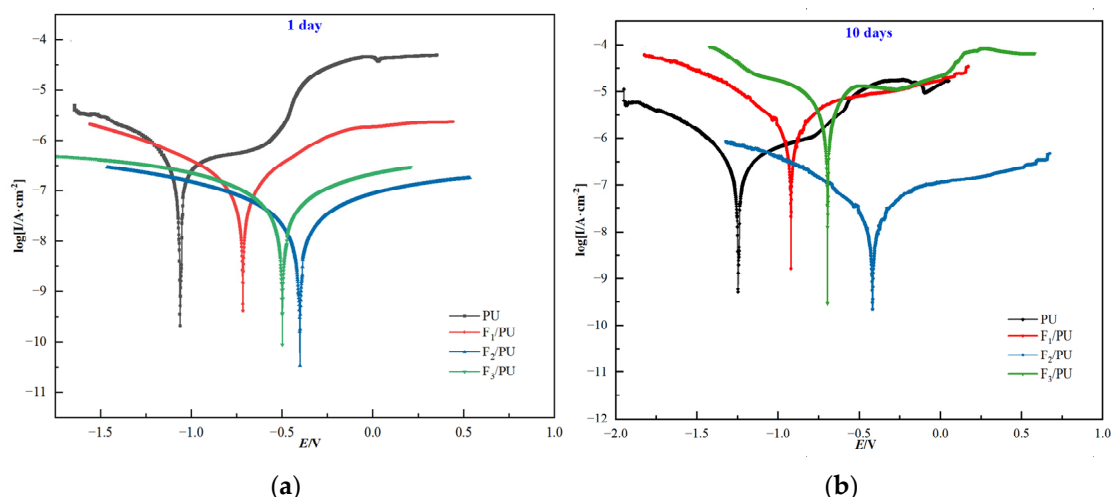


Figure 11. Polarization curves of different coatings immersed in 3.5% NaCl solution for 1 day and 10 days: (a) immersion time 1 day; (b) immersion time 10 days.

Table 4. E_{corr}, i_{corr}, and Tafel slope results of polyurethane coatings with different amounts of modified graphene oxide added in 3.5% NaCl solution.

Coating Type	E _{corr} /V	I _{corr} /(A/cm ²)	Anodic Tafel Slope β _a (mV · dec ^{−1})	Cathode Tafel Slope β _n (mV · dec ^{−1})
PU	1d: −0.62 ± 0.04	(6.35 ± 0.41) × 10 ^{−7}	128 ± 8	115 ± 7
	10d: −0.58 ± 0.03	(8.76 ± 0.32) × 10 ^{−7}	131 ± 9	119 ± 8
F ₁ /PU	1d: −0.50 ± 0.03	(2.12 ± 0.23) × 10 ^{−7}	132 ± 9	118 ± 6
	10d: −0.45 ± 0.02	(3.21 ± 0.25) × 10 ^{−7}	134 ± 10	121 ± 7
F ₂ /PU	1d: −0.38 ± 0.02	(8.54 ± 0.36) × 10 ^{−8}	135 ± 10	120 ± 8
	10d: −0.32 ± 0.01	(1.23 ± 0.15) × 10 ^{−8}	138 ± 9	123 ± 6
F ₃ /PU	1d: −0.43 ± 0.03	(1.67 ± 0.28) × 10 ^{−7}	130 ± 8	116 ± 7
	10d: −0.38 ± 0.02	(2.56 ± 0.21) × 10 ^{−8}	133 ± 8	120 ± 7

3.4. Salt Spray Performance Testing of Coatings

Specimens coated with modified graphene oxide polyurethane at varying concentrations were first subjected to a scratch test, then placed in a salt spray chamber containing a 3.5 wt% sodium chloride solution for salt spray testing. Results after 480 h of corrosion are shown in Figure 12. The figure shows that corrosion began to appear on the PU-, F₁/PU-, F₂/PU-, and F₃/PU-coated specimens at 168 h and became increasingly severe over time. At 336 h, slight corrosion appeared on the F₁/PU coating. The F₂/PU coating exhibited minor corrosion at 480 h, while the F₃/PU coating showed corrosion at 336 h. In contrast, the F₂/PU coating demonstrated significantly reduced rust spots at the scratch site and limited corrosion spread. The corrosion resistance of F₃/PU did not exceed that of F₂/PU,

indicating that excessive filler content can diminish the overall protective performance of the coating. The figure shows that the composite coating exhibits optimal corrosion resistance at an f-GO addition of 0.2%, consistent with previous test results. Specimens without modified graphene oxide additives showed the earliest corrosion onset and the poorest corrosion resistance. This confirms that incorporating modified graphene oxide enhances the coating's anti-corrosion performance, with the best results obtained at a 0.2 wt% addition.

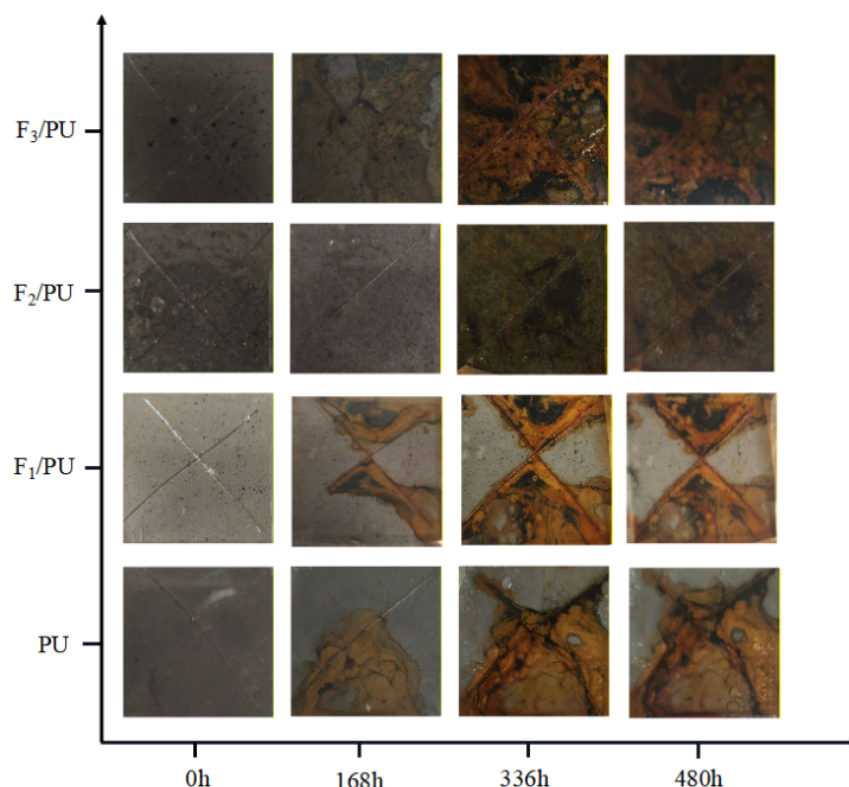


Figure 12. Salt spray test of PU-, F₁/PU-, F₂/PU-, and F₃/PU-coated specimens.

4. Analysis of Coating Corrosion Mechanisms

The corrosion protection mechanism of F/PU coatings is illustrated in Figure 13. In the corrosion protection system for Q235 steel substrates, pure polyurethane (PU) coatings cannot effectively block the penetration of corrosive media (H_2O , Cl^- , O_2) due to structural defects. Once these corrosive media penetrate the coating, they initiate electrochemical corrosion of the substrate: Fe loses electrons to form Fe^{2+} , while O_2 gains electrons in the aqueous medium to generate OH^- . These species combine to form $Fe(OH)_2$, which is subsequently oxidized to rust ($Fe_2O_3 \cdot H_2O$), ultimately leading to substrate corrosion failure. In contrast, introducing lysine-modified graphene oxide (f-GO) results in uniform dispersion of its flakes within the PU matrix, thereby forming multiple barrier pathways. This significantly extends the diffusion distance of corrosive media toward the substrate, effectively reducing the permeation rates of H_2O , O_2 , Cl^- , and other corrosive agents into the steel substrate, demonstrating excellent physical shielding effects. Furthermore, the amino and carboxyl groups in lysine molecules form hydrogen bonds and undergo partial amidation reactions with oxygen-containing functional groups on the GO surface. This enhances the interfacial bonding stability between f-GO and polyurethane segments, thereby improving the overall coating's compactness and structural stability. Simultaneously, f-GO can form cross-linking networks with carboxyl groups, amino groups, or hydrogen-bond acceptors in the PU, enhancing the mechanical strength and hydrolysis resistance of the coating.

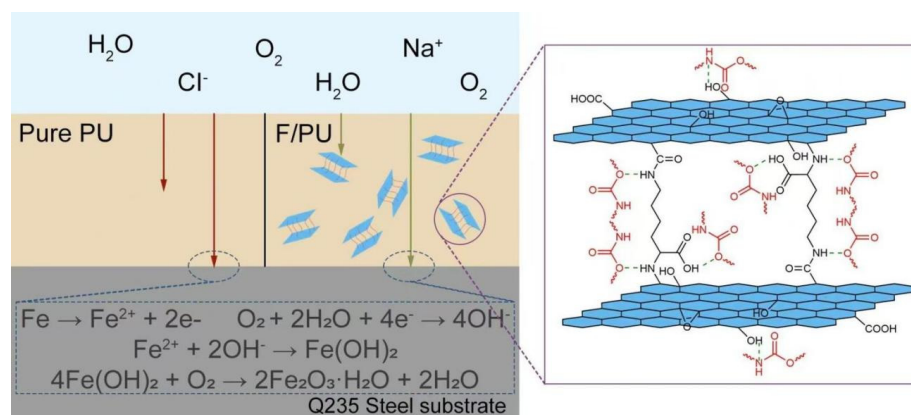


Figure 13. Schematic diagram of the corrosion prevention mechanism of F/PU coating.

In summary, lysine-modified graphene oxide effectively enhances the corrosion resistance of polyurethane coatings, providing metal substrates with longer-lasting protection in salt spray, humid, and chloride-ion environments.

5. Conclusions

This study investigated the modification of polyurethane composite coatings using lysine-modified graphene oxide. Experimental research was conducted to evaluate the corrosion resistance of modified composite coatings containing varying amounts of graphene oxide and to determine the optimal graphene oxide content. XRD, XPS, FT-IR, TGA, and SEM analyses confirmed the successful grafting of lysine onto graphene oxide, verifying its lysine modification. Polyurethane composite coatings were prepared by incorporating lysine-modified graphene oxide, and their hardness, corrosion rate, adhesion, impact resistance, electrochemical polarization curves, and impedance characteristics were systematically investigated. Results indicate that incorporating an appropriate amount of f-GO significantly enhances the coating's overall performance. As f-GO content increases, coating hardness improves, while adhesion and impact resistance initially increase before slightly decreasing. Corrosion rates decrease, polarization curves show reduced corrosion current density and positive shifts in corrosion potential, and impedance curves exhibit higher impedance values, indicating enhanced coating density and corrosion resistance. In salt spray tests, polyurethane coatings modified with lysine-functionalized graphene exhibited markedly improved corrosion resistance at scratch sites. The F₂/PU coating demonstrated optimal performance, effectively inhibiting rust propagation and delaying corrosion failure. Excessively high filler content (0.3 wt%) led to increased coating defects due to agglomeration, resulting in diminished protective performance. At 0.2 wt% f-GO content, all properties reached optimal levels: the highest coating hardness, the lowest water absorption, a significantly reduced corrosion current, and the maximum impedance, delivering the best overall protective performance. Comprehensive analysis indicates that 0.2 wt% is the optimal addition level for lysine-modified graphene oxide in polyurethane coatings, significantly improving both mechanical properties and long-term protective performance. Finally, corrosion inhibition mechanisms revealed that f-GO layers extended the penetration path of corrosive media. Lysine modification enhanced GO dispersion and interfacial bonding in PU via hydrogen and covalent bonds, while the cross-linked network between f-GO and PU increased coating density. Collectively, these effects effectively suppressed electrochemical corrosion reactions on Q235 steel substrates.

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