

Graphene oxide integrated into protective coatings against corrosion for metals and its alloys: A review

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Abstract

This review aims to discuss the effect of graphene oxide (GO) derivatives incorporated protective coatings on various metals against corrosion process in alkaline environments. These coatings increased hydrophobic character and adhesion strength between the coating and metal surface. Electrochemical measurements were used more often among all other techniques to investigate the effect of inhibitor in the polymer matrix (epoxy/silane sol). In some cases, the highest inhibition efficiency was achieved by electrochemically coating GO derivatives on the metal surface. GO derivatives prepared *via* the adsorption of Pr³⁺, Ce³⁺, and Zn²⁺ ions have revealed a good inhibition tendency for metals in acidic solution because of the exchange of Pr³⁺, Ce³⁺, and Zn²⁺ with those ions (Na⁺, Cl⁻) in the corrosion electrolyte. Further, because of the adsorption of polar groups on GO onto the surface of metals, the hydrophobicity achieved by modified GO coating limits the infiltration of corrosive ions and enhances inhibitory efficiency. And, scratches were foreseen during the manufacturing of coatings on metallic substrates, and this harms the organic coatings. The defect created by impurities developed coatings deterioration that was prone to the diffusion of corrosive substances, severely reducing the coatings' lifespan. Grafting graphene oxide with various functional groups was used to achieve self-healing behavior in organic coatings. At the defective site of the metallic substrates, self-healing by functionalized graphene oxide created dense protective coatings.

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1. Introduction

The application of organic coatings on metal substrates has been determined to protect them from corrosion in different environments. Among various coating materials, epoxy-based coatings provide a barrier effect owing to their diverse properties namely, chemical

resistance, high dimensional stability, excellent adherence to metallic substrates, and low expenditure. However, epoxy polymers possess hydrophilic hydroxyl moieties and free voids in their matrix that destroy the outstanding properties by allowing the entry of aggressive corrosive agents onto the metal surface and thus, the corrosion process gets sped up over time [1–4]. The addition of fillers/additives improves the binding properties of the coatings, enhancing the corrosion protection performance [6–12]. Amongst the additives based on shapes, for instance, spherical-like, rod-like, and sheet-like, sheet-like models drag the attention of the researchers in recent decades [13, 14].

Graphene oxide (GO), a two-dimensional carbon-based material exfoliated from graphite, grasps significant notice because of the existence of various functionalities like hydroxyl, carboxyl, and epoxy groups at their basal planes and boundaries [15–19]. The reinforcement of GO into the epoxy coatings shows an extreme resistance to corrosion as GO makes the coating impermeable to oxygen and water molecules. Unfortunately, the dispersion of GO sheets in the epoxy matrix is a difficult task because of the van der Waals forces acting between them, leading to the agglomeration of GO sheets in the epoxy system. This dispersion could be improved by covalent and non-covalent modification of GO sheets by various materials [20–25].

The present review focuses on the recent studies made on the structural modification of GO and its contribution as fillers in epoxy coatings for the protection of metals, especially in 3.5% NaCl solution. Also, to highlight the effect of GO derivatives on improving the corrosion inhibition activity by forming a protection film on the surface of metallic substrates. For which, majority of investigations have been collected and discussed that mainly focus on the use of GO as basic material for improving the protection efficiency of some metals and its alloys.

2. GO as filler for protective coatings

Graphene, a carbon allotrope, has various applications because of its fascinating electrical, optical and mechanical properties in the industrial field. Despite various advantages, it has a high tendency to undergo agglomeration and stacked sheets act like graphite having lower surface area, uniform dispersion and interfacial interaction of graphene are the most significant challenges to be satisfied. Su *et al.* [92] developed barrier films that prevent the penetration of moisture and gases for various industrial purposes. For this purpose, defectless monolayers of graphene that are impermeable to all molecules were preferred. However, it was hard to develop such a large area of graphene films appropriate for industrial application. In this work, they have produced the multilayer films by chemically reducing the GO with the help of HI and ascorbic acids. The reduced graphitic films were impermeable to all aggressive chemical substances like hydrofluoric acid. Vinothini *et al.* [93] preferred 3-amino-1,2,4-triazole-5 thiol for the functionalization of GO in the attempt of achieving a highly protective epoxy coatings for the industrial use. They reported higher values of film and charge transfer resistance and lower capacitance for fGO-epoxy coatings fabricated on the steel substrates. In addition, SECM analysis revealed lower current distribution values

compared to neat coatings at the scratched surface of the mild steel samples. Enhanced anti-corrosion activity of fGO-epoxy coated steel samples was attributed to electron donating atoms like sulfur and nitrogen. GO sheets have been found to show potential findings as promising fillers in the epoxy matrix. Table 1 & 2 shows some reported examples of protective films formed via the reinforcement of changed GO sheets with polymers in the literature for both the ferrous and non-ferrous alloys. This table lists out the studied metal, medium, additives/fillers under study (GO and its derivatives), employed experimental methods, and the results obtained from the particular investigation.

2.1. Steel and its alloys

The compatibility of GO with silane precursors was improved by integrating with tetraethoxysilane (TEOS) molecules by Li *et al.* [26]. Silanized GO sheets were treated with silane coatings and fabricated on the steel surface, which was examined by Electrochemical Impedance Spectroscopy. Results show that 0.2 wt.% of Silanized GO exhibits the highest protection properties by providing physical barrier than non-integrated coatings which controlled the intrusion of corrosion ions into the coating matrix and hindered the corrosion mechanism. Pourhashem *et al.* [27] functionalized the surface of GO with tetraethyl orthosilicate (TEOS). 0.1 wt.% SiO₂-GO sheets were mixed with epoxy matrix and coated on the steel substrates. Results reveal that the increase in pull-off adhesion property of the coating to the metal substrates and contact angle on coatings was because of the addition of 0.1 wt.% SiO₂-GO sheets. Also, salt spray test and electrochemical impedance spectroscopy (EIS) show better anti-corrosion performance with an increase in immersion time of the coated substrates in corrosion solution.

Kasaeian *et al.* [28] non-covalently functionalized GO with Benzimidazole (BIM). GO-BIM/epoxy coating fabricated on the steel surface was evaluated by electrochemical tests. Tafel results show the repression of both the cathodic and anodic branches that indicates the combined inhibition effect of Benzimidazole on steel substrate in 3.5 wt.% sodium chloride solution. Electrochemical studies reveal that the epoxy matrix containing 0.1% GO-BIM afforded superior corrosion prevention system with significantly improved inhibition and barrier performance. Di *et al.* [29] synthesized GO-ZrO₂ hybrids *via* two different silane coupling agents (GPS, APS), and the depiction of the synthesis is shown in Figure 1. The synthesized GO-ZrO₂ sheets incorporated in epoxy resin were fabricated on steel samples and were tested using electrochemical studies and FE-SEM analysis. They examined the most important factor, *i.e.*, adhesion strength, by carrying out pull-off adhesion test according to the standard ASTM D4541 and ISO 4624. Adhesion result reveals that the adhesion strength has increased for GO-ZrO₂/epoxy (12.52 MPa) coating compared to neat epoxy (10.73 MPa) coating thus, showed fewer coating delamination because of its strong adhesion to the steel surface. Also, GO-ZrO₂ sample provided excellent corrosion protection film even after 20 days of exposure to the corrosion solution, which was confirmed by FE-SEM analysis shown in Figure 2.

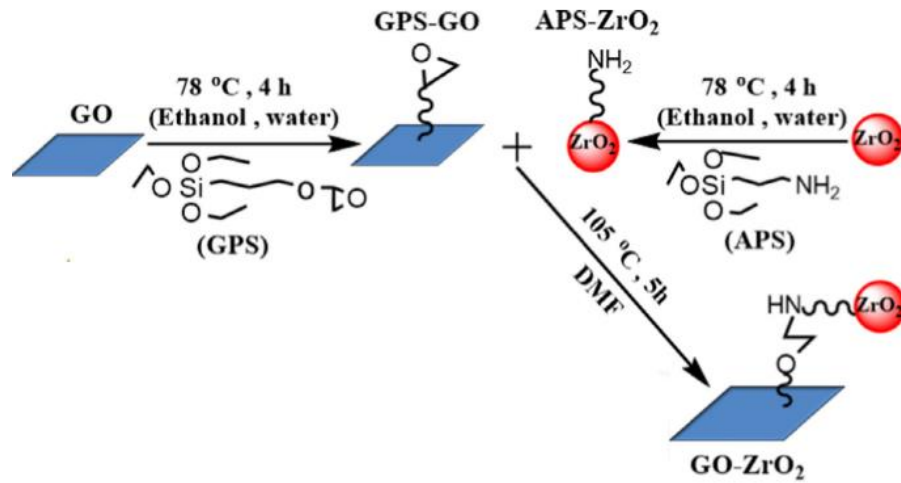


Figure 1. Scheme of the preparation of GO-ZrO₂ and its corresponding hybrid coatings [29].

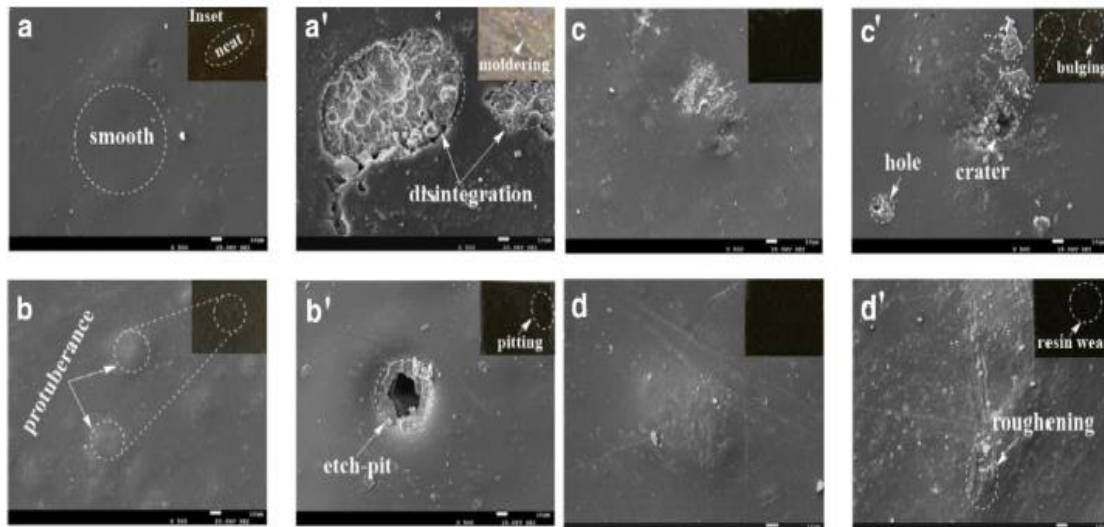


Figure 2. SEM images of electrodes about pure EP (a, a'), GO/EP (b, b'), ZrO₂/EP (c, c'), and GO-ZrO₂/EP (d, d') immersed in 3.5% NaCl solution; a–d and a'–d' corresponded to Epoxy coatings before and after immersing for 20 days, respectively [29].

Likewise, Di *et al.* [30] decorated GO surface with Fe₃O₄ particles using a silane coupling agent (APTES) and evaluated their effect on anti-corrosion behavior by preparing epoxy coatings containing different weight ratios (Fe₃O₄:GO = 1:6, 1:5, 1:4, 1:3, 1:2). Results show that the decorated GO has excellent dispersion, which helps in blocking the micro-pores produced on the steel surface and thus, Fe₃O₄ particles protect the epoxy matrix through its high-efficiency in corrosion-resistant phenomena. In other work, Parhizkar *et al.* [31] prepared 3-(Triethoxysilyl) propyl isocyanate (TEPI) reduced GO films on the steel surface at pH=4, 5, and 6 using ethanol and DMF as a solvent, individually. Also, they studied the effect of reduced GO nanosheets on the adhesion mechanism by coating GO-TEPI/epoxy on the steel substrates *via* pull-off adhesion and cathodic delamination tests.

Results reveal that the existence of isocyanate and ethoxy active agents on reduced GO decreases the coating delamination process by promoting the adhesion strength of the coatings to the steel panels and thus, fewer corrosion products were accumulated, which was evidenced by Salt spray test.

Pr^{3+} loaded GO sheets were prepared through adsorption mechanism by Samiee *et al.* [32], and evidenced the highest adsorption capacity by varying pH, adsorption dosage, temperature, and concentration of Pr^{3+} solution. They reported that the highest adsorption of Pr^{3+} on GO was achieved at pH of 7, initial concentration of 600 ppm, adsorbent dosage of 3.19 mg, contact time of 30 min, and at 35°C temperature. Electrochemical and Salt spray test results reveal that GO– Pr^{3+} sheets form a dual barrier in protecting the steel substrates from corrosion in the saline matrix. Further, GO– Pr^{3+} sheets act as self-healing agents by exchanging Pr^{3+} cations released from GO– Pr^{3+} with Na^+ ions in the corrosion medium (Figure 3). Agglomeration of GO was prevented by the surface modification with silane coupling derivatives (APTES and GPTMS) by Pourhashem *et al.* [33]. The impact of silane derivatives on the adhesion strength and contact angle were shown in Table 3. It reveals that an increase in adhesion strength and contact angle compared to neat epoxy coating were due to the hydrophobic behavior imparted by APTES and GPTMS to GO sheets. Also, silane fillers enhanced the corrosion resistance properties of neat epoxy film coated on the steel surface.

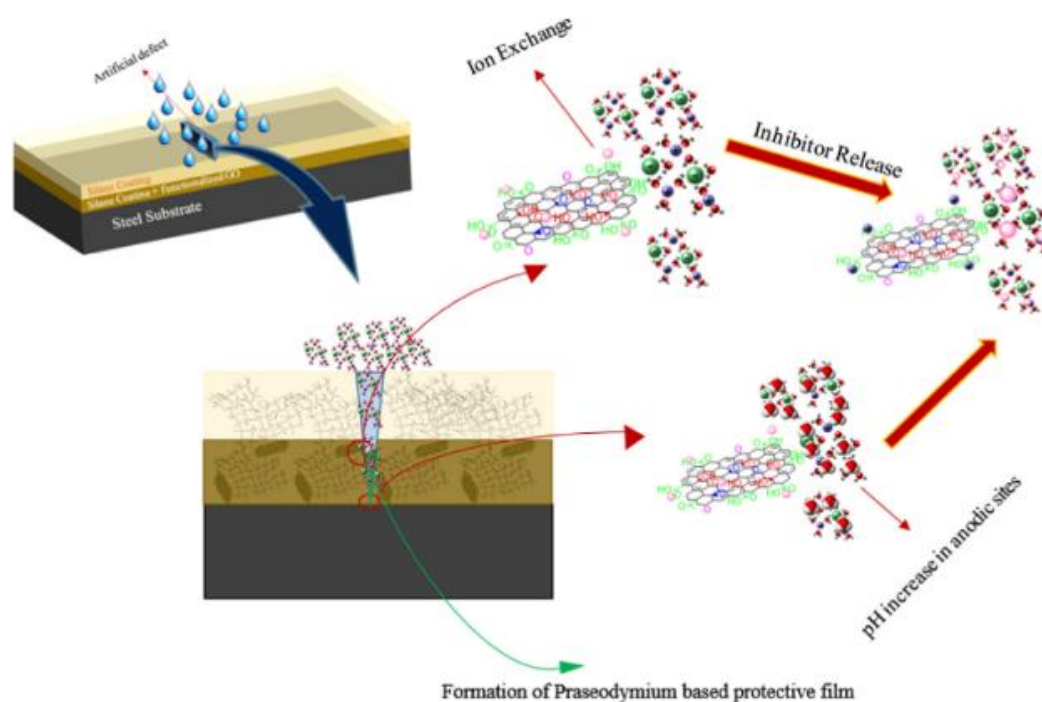


Figure 3. Schematic representation of the mechanism of self-healing ability of hybrid coatings incorporated with modified GO sheets [32].

Table 1. Some reported Graphene oxide derivatives employed in protective coatings for ferrous alloys.

Metal	Medium	Additives	Coating method	Experimental method	Results	Ref.
Mild steel	3.5 wt.% NaCl	GO-Benzimidazole/epoxy	Immersion Paint applicator	Electrochemical impedance spectroscopy(EIS) Potentiodynamic polarization Electrochemical impedance spectroscopy(EIS) Potentiodynamic polarization Salt spray test	GO-BIM provides best protection film on the active sites, especially at pH=1. GO-BIM in the epoxy matrix acts as an excellent self-repairing agent.	28
Mild steel	3.5 wt.% NaCl	GO-tetraethoxysilane (TOES)/silane sol	Drop-casting	Electrochemical impedance spectroscopy(EIS) FE-SEM	0.2 wt.% GO-TOES shows good dispersion and compatibility in silane matrix resulting in improved protection behavior.	26
Carbon steel	3.5 wt.% NaCl	GO-tetraethyl orthosilicate (TEOS)/epoxy	Air spray method	Electrochemical impedance spectroscopy(EIS) Potentiodynamic polarization nergy dispersive X-ray spectroscopy (EDS) Pull-off Test Salt spray test	0.1 wt.% GO-SiO ₂ enhances adhesion strength <i>via</i> strong interfacial interaction with the epoxy matrix.	27
Mild steel	3.5 wt.% NaCl	GO-3-(Triethoxysilyl) propyl isocyanate (TEPI)/epoxy	Film applicator	Electrochemical impedance spectroscopy(EIS) Potentiodynamic polarization Pull-off Test Cathodic disbonding test Salt spray test	Extreme corrosion protection rate for GO-TEPI sample because of excellent barrier activity. Also, reduction in coating delamination diameter was observed.	31
Mild steel	3.5 wt.% NaCl	GO-Pr ³⁺ /silane sol	Dip-coating method	Electrochemical impedance spectroscopy(EIS) Salt spray test	GO-Pr ³⁺ induces self-healing property at the defected sites.	32

Metal	Medium	Additives	Coating method	Experimental method	Results	Ref.
Mild steel	3.5 wt. % NaCl	GO-ZrO ₂ /epoxy	Air spray method	Electrochemical impedance spectroscopy(EIS) Pull-off Test	2 wt.% GO-ZrO ₂ 2D sheets in epoxy matrix not only improve barrier effect by capping the minute pores created in the active sites but also enhance the adhesion strength of the coating.	29
Mild steel	3.5 wt. % NaCl	GO-APTES & GPTMS(silane coupling agents)/epoxy	Air spray method	Electrochemical impedance spectroscopy(EIS) Pull-off Test Contact angle measurements Salt spray test	0.1 wt.% GO-APTES & GPTMS increases adhesion strength and contact angle on the epoxy coatings. Also, promotes corrosion inhibition action by forming a protective film.	33
Mild steel	3.5 wt. % NaCl	GO-Fe ₃ O ₄ /epoxy	Air spray method	Electrochemical impedance spectroscopy(EIS) FE-SEM	2 wt.% GO-Fe ₃ O ₄ magnetic hybrid sheets provide high efficiency anti-corrosion film and it finds application in wastewater treatment.	30
Mild steel	3.5 wt. % NaCl	GO-Ce ³⁺ /epoxy	Film applicator	Electrochemical impedance spectroscopy(EIS) Potentiodynamic polarization Pull-off Test Cathodic disbonding test Salt spray test	Release of Ce ³⁺ ions is responsible for the creation of protection film at the defected part.	34
Mild steel	3.5 wt. % NaCl	GO-Zn ²⁺ /epoxy	Paint applicator	Electrochemical impedance spectroscopy(EIS) Potentiodynamic polarization Pull-off Test Cathodic disbonding test Salt spray test	GO-Zn ²⁺ enhances compatibility and imparts active/passive barrier effects simultaneously to epoxy matrix at acidic conditions.	35

Table 2. Some reported Graphene oxide derivatives employed in protective coatings for non-ferrous alloys.

Metal	Medium	Additives	Coating method	Experimental method	Results	Ref.
Copper	3.5 wt.% NaCl	GO-Melamine/epoxy	Dipping	Electrochemical measurements	By evaluating the different concentrations of inhibitors, highest <i>IE</i> % of 90.83% was observed at 1.0 mg/mL (25°C)	36
Copper	3.5 wt.% NaCl	N-doped GO/rGO/epoxy	Electrophoretic deposition process	Potentiodynamic polarization studies	Comparing with GO, rGO & N-doped GO, good inhibitor efficiency was observed for rGO. Reason is catalytic activity towards oxygen reduction of N-GO	37
Copper	3.5 wt.% NaCl	GO-Zn/epoxy	Wire bar coater	Open circuit potential, Electrochemical impedance spectroscopy, Electrical equivalent circuits	Zinc-rich-epoxy layer shows better anticorrosion activity than neat epoxy in marine system	38
Copper foil	3.5 wt.% NaCl	GO- <i>p</i> -phenylenediamine/epoxy	Electrophoretic deposition	Potentiodynamic polarization studies	Efficiency factor was found to be 0.284, which was higher than the reported literature values of GO systems	39
Copper	3.5 wt.% NaCl	GO-Silane derivative/epoxy	Electrophoretic deposition	Electrochemical Impedance	Small-sized fGO show better corrosion protection than large-sized fGO	40
Copper	3.5 wt.% NaCl	GO-PVPBM (poly 4-vinylpyridine-co-butyl methacrylate)/epoxy	Electrophoretic deposition	Thermo-gravimetric analysis, potentiodynamic polarization measurements, and electrochemical impedance spectroscopy	95.4% efficiency was reported by modified GO coating on Cu	41

Metal	Medium	Additives	Coating method	Experimental method	Results	Ref.
Copper	3.5 wt.% NaCl	GO	Electrophoretic deposition	Electrochemical measurements	rGO shows better efficiency (54.40%) than GO coated Cu, and the corrosion rate was 0.18 mm/year	42
Copper	3.5 wt.% NaCl	GO	Electrophoretic deposition	Tafel analysis and electrochemical impedance spectroscopy	At the deposition voltage of 5 V and time duration of 10 s, inhibition efficiency was in the increasing order: 3 mg/mL < 1 mg/mL < 2 mg/mL ss	43
Copper	3.5 wt.% NaCl	GO-Polymethylmethacrylate/epoxy	Drop casting	Electrochemical measurements	GO-PMMA coating shows high efficiency (99.27%) at 100 h of immersion	44
Copper	3.5 wt.% NaCl	GO-polymer composite (PC) (Polymeric isocyanate crosslinked with hydroxy functional acrylic Adhesive)/epoxy	Electrophoretic deposition	Potentiodynamic polarization studies, Electrochemical impedance spectroscopy	PCGO reduced the corrosion rate of Cu metal by 3 times compared to neat GO	45
AZ31Magnesium alloy	Simulated Body Fluid (SBF)	GO-Hydroxyquinoline-MgO/Mg ₃ PO ₄ /epoxy	Immersion	Potentiodynamic polarization and electrochemical impedance spectroscopy	HQ+GO/MgO and HQ+GO/Mg ₃ PO ₄ show good corrosion resistance than HQ+GO	57
AZ61 Mg alloy	Simulated Body Fluid (SBF)	GO-MgO/epoxy	Selective laser melting	Electrochemical studies	GO-MgO composite have good inhibitive effect for AZ61	58
AZ31B Mg alloy	Simulated Body Fluid (SBF)	GO-Chitosan/heparinized (Chi/HGO) multilayer/epoxy	Immersion	Electrochemical studies	Chi/HGO multilayer not only decreases the rate of corrosion but also improves the biocompatibility	59

Metal	Medium	Additives	Coating method	Experimental method	Results	Ref.
AZ31 Mg alloy	3.5 wt.% NaCl	GO-Carboxymethyl cellulose/epoxy	Immersion	Electrochemical Measurements	Efficiency of CeO ₂ /(PEI/CMC-GO) was 90%	60
AZ91E Mg alloy	Simulated Peritoneal Fluid (SPF)	GO-polythreonine /epoxy	Electrochemical deposition method	Electrochemical impedance spectroscopy and potentiodynamic polarization experiments	The corrosion resistance and bioactivity, like gastrectomy applications, were also improved	61
AZ31 Mg alloy	3.5 wt.% NaCl	GO-8-hydroxyquinoline (8-Hq) /inorganic coating (IC) (GO/8-Hq/IC)/epoxy	Plasma Electrolytic Oxidation and Dip-chemical coating	Electrochemical Measurements	Good anti-corrosion coating has been achieved in saline medium	62
AZ91D Mg alloy	3.5 wt.% NaCl	GO-Acrylamide /acrylic acid/epoxy	Wet film applicator	Potentiodynamic polarization and electrochemical impedance spectroscopy	High Efficiency (99.8%) reported and I_{corr} value is 650 times lower than uncoated alloy	63
AZ91 Mg alloy	3.5 wt.% NaCl	GO-Triethanolamine (TEA)/epoxy	Electro deposition	Electrochemical tests, immersion experiments and quantum chemical calculation	The efficiency of GO-TEA is higher (93.026%) than the TEA coating (86.293%) on the alloy surface	64
AZ91D Mg alloy	0.1 M NaCl	GO/zinc–aluminum layered double hydroxide/epoxy (GO/Zn–Al LDH)	One-step facile hydrothermal crystallization	Potentiodynamic polarization measurements	$IE\% = \text{GO/Zn–Al LDH} > \text{Zn–Al LDH}$ on AZ91D Mg alloy	65
Mg	0.1 M NaCl and 0.1 M Na ₂ SO ₄	GO/SiC/epoxy nano-composites	Linear Electrodeposition technique	Tafel polarization measurements	GO/SiC shows good corrosion resistance in both NaCl and Na ₂ SO ₄ aqueous medium	66

Table 3. Adhesion strength and contact angles of silane derivatives modified GO epoxy coatings.

Samples	Adhesion strength (MPa)	Contact angle (°)
Neat epoxy	8.5±0.5	73.1±2
GO/epoxy	11±1	70.7±2
GO–A/epoxy	17.7±0.5	82.9±1
GO–G/epoxy	16.5±1	87.9±1

2.2. Copper and its alloys

The corrosion resistivity of GO has been improved by functionalizing with melamine by Li *et al.* [46]. The copper substrate was in melamine treated GO (MGO) at different concentrations. The immersion test reveals that smooth surface was formed after coating. Electrochemical results show an improvement in the corrosion-resistant property of neat GO after modification with melamine on Cu substrate. 1 mg/mL of MGO coated on the substrate shows a high efficiency of about 90.83% in 3.5 wt.% NaCl solution. Likewise, the corrosion resistance of GO was improved by reducing with zinc by Ge *et al.* [47]. The reduced GO/epoxy coatings were fabricated on Cu substrate through wire bar coater, which enhanced the sacrificial anode protection and improved barrier property of waterborne epoxy coatings. The equivalent electric circuits for epoxy, epoxy-Zn, epoxy-G, epoxy-Zn-G, and epoxy-stack are shown in Figure 4. Electrochemical studies prove the long-time corrosion resistance of the changed epoxy coatings.

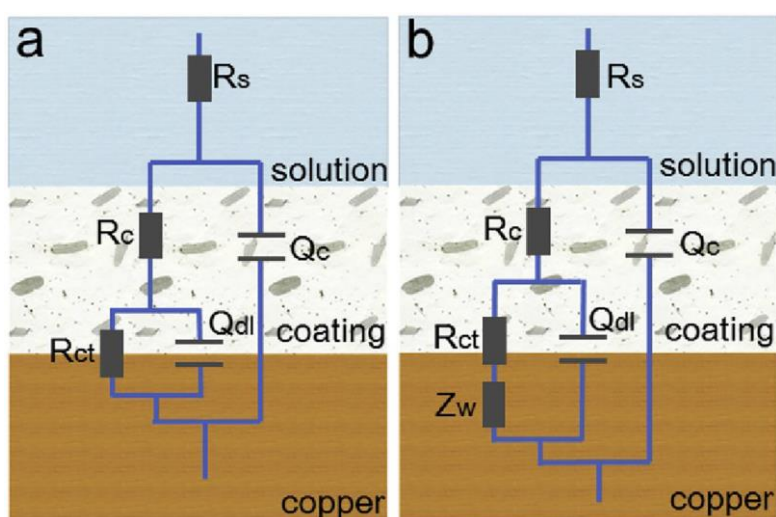


Figure 4. The equivalent electric circuits for epoxy, epoxy-Zn, epoxy-G, epoxy-Zn-G, and epoxy-stack [47].

2D structured GO was changed by *p*-phenylenediamine (PPD) and applied on the surface of Copper by Hwang *et al.* [48]. GO-PPD was coated on the metal substrate by the cathodic electrophoretic deposition (EPD) method. Field emission scanning electron

microscopy analysis showed the smooth cracks on the surface because of coatings. Anti-corrosion test shows that GO-PPD/epoxy coatings act as an excellent corrosion protection film because of their anodic barrier effect on the copper surface in various corroding environments. Ollie *et al.* [49] synthesized nitrogen-doped GO to prepare anti-corrosion coatings. The prepared N-GO was coated on copper metal by an electrophoretic deposition method. Morphological analysis shows the formation of the least hydrophilic and improved adhesion strength of the coatings on Cu surface. Raza *et al.* [50] reported the silane functionalized GO derivative for the corrosion prevention of copper. In this study, they prepared silane functionalized-GO both as flakes and powder (FGO & PGO) (Figure 5) and coated effectively on the copper surface by an electrophoretic deposition method. GO and silane-functionalized GO coatings were evaluated by Atomic Force microscopy, Fourier transforms infrared spectroscopy, and Raman spectroscopy. Results reveal that PGO-coated Cu substrates show better corrosion resistance when compared with FGO-coated Cu.

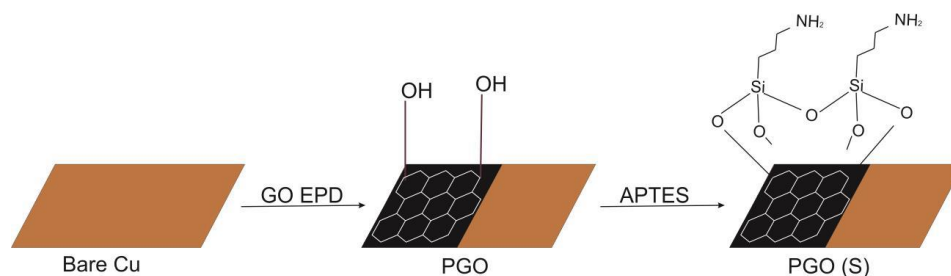


Figure 5. Schematic showing surface functionalization of GO by APTES.

The corrosion resistance and mechanical properties of copper were improved by Kumari *et al.* [51]. For which, they prepared reduced GO (RGO)-poly (4-vinylpyridine-co-butyl methacrylate) (PVPBM) composite coatings and applied by electrophoretic deposition (EPD) method on the metal surface. Thermo-gravimetric analysis (TGA) shows that the thermal stability of RGO-PVPBM composite coating was higher when compared to PVPBM. Also, results highlighted the enhanced micro scratch adhesion and crack propagation resistance. Calculated corrosion inhibition efficiency and corrosion rate from electrochemical studies were 95.4% and 0.02 mm/year, respectively. Kiran *et al.* [52] reported an inexpensive transparent GO coating for Cu. GO prepared by Modified Hummer's method was coated on metal through an electrophoretic deposition process and it was finally reduced by sodium borohydride. The chemical characterization was done by FE-SEM, FT-IR, Raman spectroscopy, UV-Vis, and XRD analyzes. Results reveal 2D band GO coatings on the substrate. Electrochemical data shows that the coating reduced the corrosion rate appreciably.

Investigation of corrosion-resistant behavior of neat GO was done for Cu Raza *et al.* [53]. The electrophoretic deposition method was used to coat GO on the metal surface. Increased voltage shows the increased thickness of the coatings. And, surface morphologies reported that the pores and cracks on the coating were reduced upon the addition of GO. EIS and Tafel analysis reveals the higher efficiency of GO/epoxy coatings were achieved

through EPD method. Qi *et al.* [54] synthesized a Nano-composite material in solution-processable polymer using PMMA grafted GO for corrosion prevention. Grafted polymethylmethacrylate (PMMA) was brushed on GO through Atom transfer radical polymerization (ATRP) on copper substrate. FT-IR, TGA, GPC, XPS, SEM, TEM, and AFM analyses were employed for characterization. Electrochemical measurement reveals that corrosion current density was decreased by 81% PMMA-g-GO coating on the metal surface. Coatings prepared using functionalized GO showed good corrosion resistance for Cu up to 100 hours in the corrosive medium.

A GO-polymer composite coating was fabricated by Singh *et al.* [55] for the reduction of corrosion and oxidation processes on the copper substrates. Polymeric isocyanate cross-linked with hydroxy-functional acrylic (PIHA) was treated with GO to convert into GO/PIHA (GOPC) composite coatings and were coated on copper substrate by Electrophoretic Deposition method. The water repellent protective organic layer formed by the coating enhanced corrosion resistivity. Results reveal that oxidation and corrosion resistance of GOPC coatings were improved, and high protective shielding was formed on the substrates. Vinothkumar *et al.* [56] improved the corrosion resistance activity of copper by preparing and coating the substrates with 3-amino-5-mercapto-1,2,4-triazole (AMTa) functionalized GO by Electropolymerisation Method. AMTa+GO coatings were characterized by Fourier Transform Infrared (FT-IR) spectroscopy, Raman spectroscopy, X-ray photoelectron spectroscopic (XPS) analysis, and Energy Dispersive X-ray (EDX) spectroscopy. Results reveal that an effective corrosion-resistant coating was formed on Cu surface.

2.3. Magnesium and its alloys

Wen *et al.* [67] reported the functionalization of GO with hydroxyapatite for Magnesium alloy. 1 wt.% of HA/GO synthesized by sol-gel method and was applied on the substrate surface by a one-step micro-arc oxidation method. The pores of the surface of Mg alloy were sealed by the HA/GO composites, which was resulted from SEM results. From high polarization resistance and low corrosion current density, it was evident that an effective barrier was developed by the coating. Results reveal that HA/GO coating enhanced the corrosion resistance property of the alloy to a good extent.

Zhao *et al.* [68] used Plasma electrolytic oxidation coating method for coating GO on the surface of AZ31 Magnesium alloy. Two-step electrolytic oxidation coating was done by using constant current followed by constant voltage. Uniform coating having 2 g/L GO was achieved with PEO method, and very less porosity was observed. Electrochemical studies reveal the improvement in corrosion resistance with the increase in concentration which readily increases the carbon content.

A multi-layered self-healing corrosion-resistant coating was prepared by Fan *et al.* [69] for Magnesium alloy. Cerium-treated Mg was fabricated with poly ethylene imine, GO, and polyacrylic acid as layer by layer to get Ce(IV)/PEI/GO/(PEL/PAA). Electrochemical study results reveal that the coating with GO shows better anti-corrosion properties than the

coating without GO. Further, better self-healing ability was achieved by increasing the number of layers to 10 layers on the substrate. Catt *et al.* [70] synthesized polyethylenedioxythiophene and nano-GO composite coating on Magnesium for medical application. Dexamethasone, an anti-inflammatory steroid was implanted with the above composite, and the produced polymer PEDOT/GO/Dex was coated on the metal surface. Electrochemical impedance spectral results show that the corrosion resistance of the metal has been increased well compared to the bare metal. Hydrogen evaluation tests also supported the reports. Further, bi-functional nature and anti-inflammatory behavior were revealed by this study.

A two-layer coating of nano-silica/GO was developed by Bakhsheshi *et al.* [71] Nano-SiO₂ was coated by physical vapor deposition and GO by dipping methods. Electrochemical studies explained better anti-corrosion activity of GO incorporated coating, and disc diffusion antibiotic sensitivity testing for antibacterial activity against *Streptococcus mutans* revealed good results. Perfluorinated polysiloxane–GO composite was synthesized to evaluate the anti-corrosion property of the coating on AZ31 Mg alloy by Ikhe *et al.* [72] PPFS was prepared from 1H,1H,2H,2H-perfluorooctyltriethoxysilane and treated with GO to produced (PPFS/GO) (Figure 6). The effect of PPFS/GO coating on the corrosion protection of AZ31 was confirmed by electrochemical impedance spectroscopy and potentiodynamic polarization tests in 3.5 wt.% NaCl which confirms the high corrosion resistant property and promising coating for Mg and its alloy.

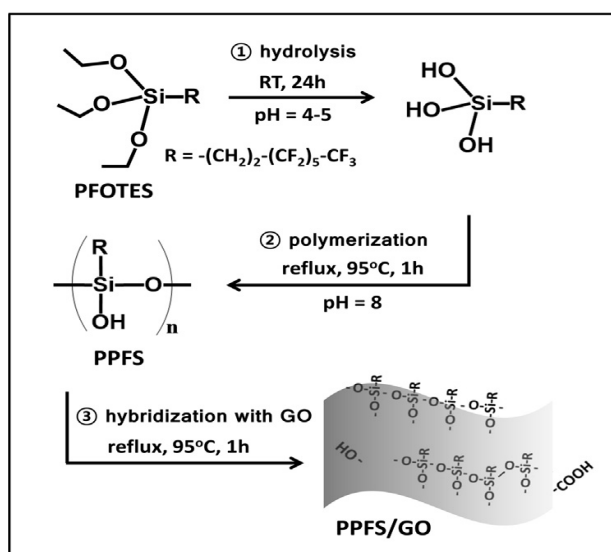


Figure 6. Schematic representation of the synthesis of PPFS/GO.

Fernández *et al.* [73] synthesized reduced GO by electrochemical and chemical methods on the surface of Mg-disc in alkaline medium. Bovine serum albumin (BSA) was coated on a passivated metal surface to generate positive changes for the anchoring of GO. The electrochemical impedance spectroscopy and potentiodynamic polarization studies ensure good corrosion resistivity on Mg alloy. SEM images also confirm the increment of

the corrosion resistance on the samples. GO/reduced GO (rGO)-polyvinyl alcohol (PVA) composites were developed for Mg alloy in an alkaline medium by Chu *et al.* [74]. The GO/rGO-PVA hybrid coatings were fabricated on the surface of alloy by spin-coating technology. Oxygen-containing groups have been removed by the coating. The intrinsic cathodic reduction reaction and the galvanic corrosion behavior between alloy and GO acted as a barrier for Mg alloy. From the electrochemical studies, it is clear that the coating reduced the corrosion rate to good extent.

Gao *et al.* [75] synthesized layer by layer of chitosan-functionalized GO (GOCS) bioactive materials to improve corrosion resistivity and biocompatibility of magnesium alloy. The 16-phosphonyl-hexadecanoic acid pre-treated Mg alloy was immersed in GOCS and heparin to get Mg-GOCS/Hep coating. Corrosion was reduced considerably by heparin dense coating (Figure 7). The coating had good blood compatibility and also inhibited platelet adhesion and activation. Jin *et al.* [76] prepared a nano NH_2 -reduced GO (NGO) and NGO/epoxy resin for coating on Mg alloy, and the effect of coating on anti-corrosion behavior was reported. NGO was prepared by treating NH_3 with GO, which was treated with epoxy resin and coated by wet film applicator. Electrochemical measurements show good corrosion resistance in 0.3 wt.% NaCl. The coating showed not only high corrosion inhibition efficiency, but also proved good self-healing property on the Mg alloy substrate.

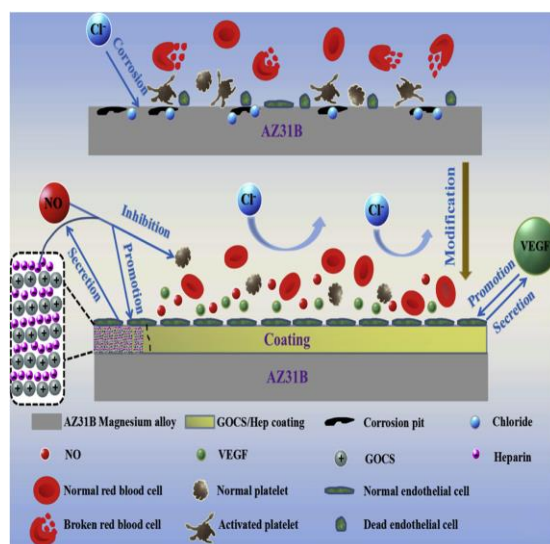


Figure 7. Mechanism of GOCS/Hep multilayer coating on the corrosion resistance and biocompatibility of the magnesium alloy surface.

2.4. Aluminum and its alloys

Zhu *et al.* [77] prepared GO–polyvinyl butyral composite coatings for improving the anti-corrosion property of Aluminum substrate. The SEM analysis showed the uniform distribution of the composite coating and EIS measurements in 3.5 wt.% NaCl solution revealed the significant anticorrosive effect of the coating on Aluminum surface. The frequency impedance modulus value reached $10^7 \Omega \cdot \text{cm}^2$, which was a three-fold increase

compared with bare metal. The potentiodynamic studies show three orders of magnitude decrease in corrosion current density (I_{corr}) than bare aluminum. The coating was effective in protecting the substrate up to 1200 h, and an increase in contact angle from 81° to 107° shows the boost up in hydrophobic behavior of the coating. The cross square adhesion test reveals the well-adhered nature of GO/PVB coatings on Aluminum substrate.

Yuan *et al.* [78] prepared Polyaniline/reduced GO (PANI/RGO) by reducing Polyaniline/GO (PANI/GO) with hydrazine. Epoxy coatings were loaded with PANI – 2, 5, and 8 wt.% GO and fabricated on Aluminum alloy. SEM images of modified composites show the retardation of the agglomeration of PANI due to in-situ polymerization between GO and PANI. Electrochemical impedance spectroscopy (EIS) reveals the improvement in passivation of the metal even after the immersion period of 30 days, stable barrier effect was shown by epoxy/PANI-5 wt.% RGO when compared to epoxy/PANI-8 wt.% RGO and epoxy/PANI-2 wt.% RGO. They proposed that the amount of active PANI has been increased by RGO, and this increased the ability to form a protective barrier on the Alloy surface.

Zhao *et al.* [79] reported the layer-by-layer self-assemble films comprising of formation and deposition of polyelectrolytes (PEI & PAA) with and without GO were fabricated on Aluminum alloy. The surface morphology of LBL films was investigated, indicating that GO has provided smooth and uniform self-assembling films that might greatly impact the anti-corrosion properties of layers. EIS studies reveal that the inhibition efficiency of the protective film decrease in the order: LBL-2 > LBL-1 > Ce-CF in protecting the metal from corrosion, and this stands as evidence for the reaction of LBL and GO. The LBL self-assembled film modified by GO showed the inhibition efficiency of 99.8% in 3.5 wt.% NaCl solution and has to wear resistance with an efficiency of 96% when dragged to a distance of 400 mm.

Badr *et al.* [80] improved the corrosion resistance property of Aluminum alloy by coating with GO and reduced GO. Reduced GO was prepared by electrochemically reducing GO in cyclic voltammetry in 0.1 M K_2HPO_4 solution. Results reveal that GO and reduced GO forms uniform layered structures on the substrate surface. Also, oxygenated functional groups on GO have been reduced upon electrochemical reduction. EIS studies indicate that the reduced GO has an adverse effect on the anti-corrosion properties of GO in 3.5 wt.% NaCl solution by increasing the thickness of the coating on the alloy surface.

Naghdi *et al.* [81] deposited Ca^{2+} (from CaCO_3) intercalated GO by cathodic electrophoretic deposition on aluminum alloy. EDAX analysis showed a decrease in oxygen/carbon ratio after reduction. The water contact angle increased with an increase in different annealing temperature (50° – 200°C) which stands as a reason for enhanced anti-corrosion properties in saline media. Further, the increase in temperature of the coated samples improved the hydrophobicity of the Aluminum substrate. SEM analysis was employed for coated samples after and before immersion in 3.5 wt.% NaCl solution showed fewer cracks and pits on the surface of GO-coated samples when compared with the bare sample.

Titanium dioxide incorporated GO ($\text{TiO}_2\text{-GO}$) was prepared using 3-aminopropyltriethoxysilane (APS) by Liu *et al.* [82]. $\text{TiO}_2\text{-GO}$ was modified by γ -(2,3-epoxypropoxy) propyltrimethoxysilane (GPTMS), dispersed in epoxy resin, and fabricated on Aluminum substrate. Results reveal that the corrosion current density of GPTMS modified $\text{TiO}_2\text{-GO}$ /epoxy composite decreases by two-fold than neat GO/epoxy composite in basic medium. Also, GPTMS/ $\text{TiO}_2\text{-GO}$ modified sample improved the adhesion strength from 57% to 86%. GPTMS has improved the dispersion of GO in epoxy matrix thus, cross-linking and adhesion strength has been promoted in GPTMS/ $\text{TiO}_2\text{-GO}$ /epoxy coated sample than neat GO coated sample.

3. Mechanism of corrosion protection by GO-based coatings in alkaline media

During the fabrication of coatings on the metallic substrates, scratches were foreseeable and that causes harm to the organic coatings. Impurities that are present in the coatings and cracks produced owing to aging of coatings were the chief causes for the flaw in the coatings. These defected areas were prone to the diffusion of corrosive media and strictly slow down the life of coatings. Hence, self-healing of the damage was considered as the hot area of research nowadays. Self-healing behavior of the organic coatings could be achieved by grafting graphene with various functional groups. Self-healing by functionalized graphene produced dense protecting film at the defected site of the metallic substrates. Based on the type of film formation, there are two types: catalytic passivation film, and adsorption film.

3.1. Catalytic passivation film

The redox catalysis of various functionalities grafted over graphene is generally referred to as catalytic passivation. This resulted in the creation of passivation film on the metallic surface. In the study of polyaniline coatings, Aminobenzenes are the groups that are reported of this kind. It was revealed from the electrochemical study that anticorrosion activity of polyaniline was not attributed by polymer coatings such as polystyrene and epoxy coatings. They assumed that this was associated to the catalytic activity of polyanilines that pronounced the development of protective films on the surface of the metal [83]. Further the study on catalytic passivation was briefed out by the research on the development of aniline based substances. *N,N'*-bis(4'-aminophenyl)-1,4-quinodiimine and its derivatives were synthesized by the oxidation of aniline with *p*-phenylenediamine in the presence of acidic aqueous medium. 1,4-phenylenediamine derivatives were obtained by the reduction of 1,4-quinodiimine compound. The obtained products were further subjected to the synthesis of aniline oligomers [84]. This stands as the basis for the structural modification of graphene with aniline. 1,4-phenylenediamine and aniline were reacted in the presence of ammonium persulfate, an oxidizing agent. An polyimide resin was formed as the result of reaction of amino-terminated trimer and 4,4'-(4,4'-isopropylidiphenoxy)bis(phthalic anhydride). This polyimide resin catalysed the development of passivation film on the steel surface which was mainly attributed to redox catalytic behavior of amino-terminated trimer present in the polyimide resin [85]. The incorporation of graphene in polyimide resin enhanced the

shielding effect graphene in addition to passivation of amino-terminated aniline trimer. It is obvious from the literature that Graphene/polyimide coatings were prepared *via in situ* polymerization. The raw materials for this polymerization are GO and amino-terminated aniline trimer [85]. In other work, the aniline-based trimer was incorporated onto GO followed by the addition of aniline trimer-GO to resin as the additive fillers. Thorough survey on the literature revealed that triethoxysilylpropyl isocyanate and aniline trimer were used for synthesise of silylated aniline trimer. This trimer bounds to GO by p-p conjugation under ultrasonic conditions. Enhanced dispersibility of GO in epoxy resin was observed and silylated aniline trimer promoted the formation of protective film made of Fe₂O₃ and Fe₃O₄ on the metallic surface [86].

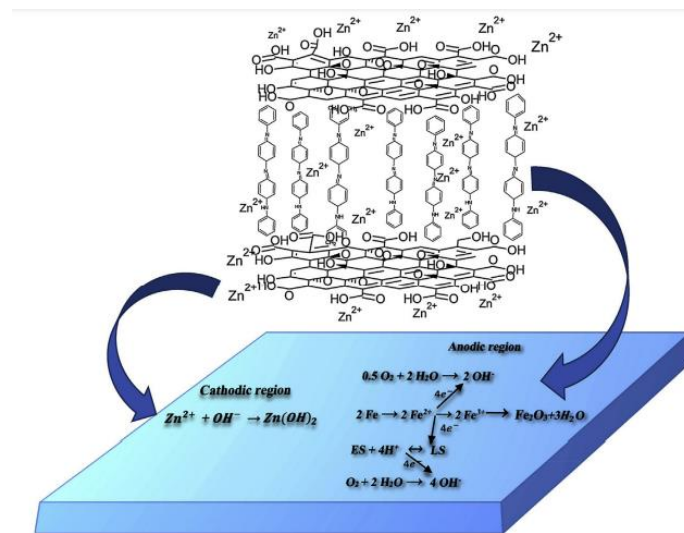


Figure 7. Formation mechanism of passivation film catalyzed by GO/polyaniline/zinc composites [91].

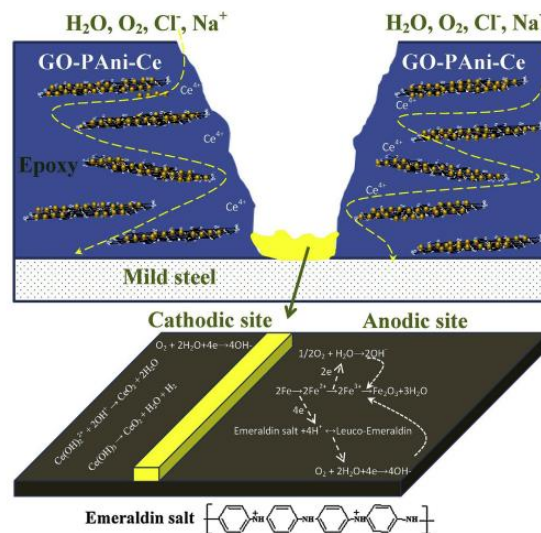


Figure 8. Polyaniline and cerium oxide promote the formation of passivation film in the defect region of the coatings [91].

The synergistic effect with zinc oxide or cerium oxide enhances the redox catalytic passivation of aniline compounds. In the literature, myristic acid was grafted on the surface of GO, and GO/polyaniline/zinc composite was prepared by using myristic acid-modified GO, sodium lauryl sodium, ammonium persulfate, aniline and zinc nitrate as raw materials. The author prepared the GO/polyaniline/cerium oxide compound in a similar manner [87]. In the composite material, zinc (or cerium oxide) was electrostatically or cationically conjugated to molecules of polyaniline. The synergistic effect of polyaniline and zinc oxide or cerium oxide made it easier to form a high-quality film on the steel surface (Figures 7 & 8), and the corrosion of the steel substrate was noticeably concealed.

3.2. Adsorption film

The corrosion-inhibiting adsorption film mainly involves the combination of some corrosion-inhibiting groups grafted with graphene and metal atoms or ions to form an adsorption film on the metal surface. It combines the enhanced protection of graphene and an adsorption film that inhibits corrosion. As mentioned in the previous section, some studies have shown that aminobenzene promotes the formation of passivation films on metal surfaces. There are also some publications showing that aminobenzenes forms a corrosion-inhibiting film on the metal surface. However, whether aminobenzenes enhanced the formation of a passivation film or a corrosion-inhibiting film on the metal surface has not yet reached a consensus. Regarding the latter, the following research was conducted. Aminoazobenzene or diaminobenzene was grafted on the surface of GO, and the modified graphene inhibited the corrosion of the steel substrate by forming an adsorption protective film on the steel surface. Both of them are mixed corrosion inhibitors mainly to inhibit the cathodic reaction, and the corrosion inhibition effect of aminoazobenzene was found to be better than that of diaminobenzene [88].

Azole compounds are commonly used as corrosion inhibitors, such as metronidazole and benzotriazole. Metronidazole has a tendency to bond with carboxyl groups, so GO must be carboxylated with maleic anhydride. Experiments have shown that in epoxy resin, modification of metronidazole promoted dispersion of graphene, and metronidazole groups act as corrosion inhibitors to form an adsorption film on damaged parts of the coating, slowing down corrosion of the coating. metallic matrix. B-Cyclodextrin could load and control benzotriazole (Figure 10). First loaded b-cyclodextrin on the surface of the graphene and placed benzotriazole on b-cyclodextrin. When corrosion occurred, an acidic or alkaline environment was generated locally. B-Cyclodextrin released benzotriazole molecules in an acidic or alkaline environment. Benzotriazole was adsorbed on the metal surface to form a film that prevented further corrosion and has a certain self-healing effect [89]. Some natural biological extracts also act as corrosion inhibitors. The major components of nettle leaf extract are histamine, serotonin and quercetin. Researchers prepared modified GO by directly reacting to nettle leaf extract with GO under stirring [90]. The modified graphene and zinc ions act synergistically to form an adsorption protective film and zinc hydroxide protective film on the surface of the steel substrate. Both the cathodic and anodic reactions

on the surface of the steel substrate are suppressed [91]. The GO modified by the nettle leaf extract showed a self-healing effect in the damaged areas of the epoxy coating and the polyaniline coating (Figure 11).

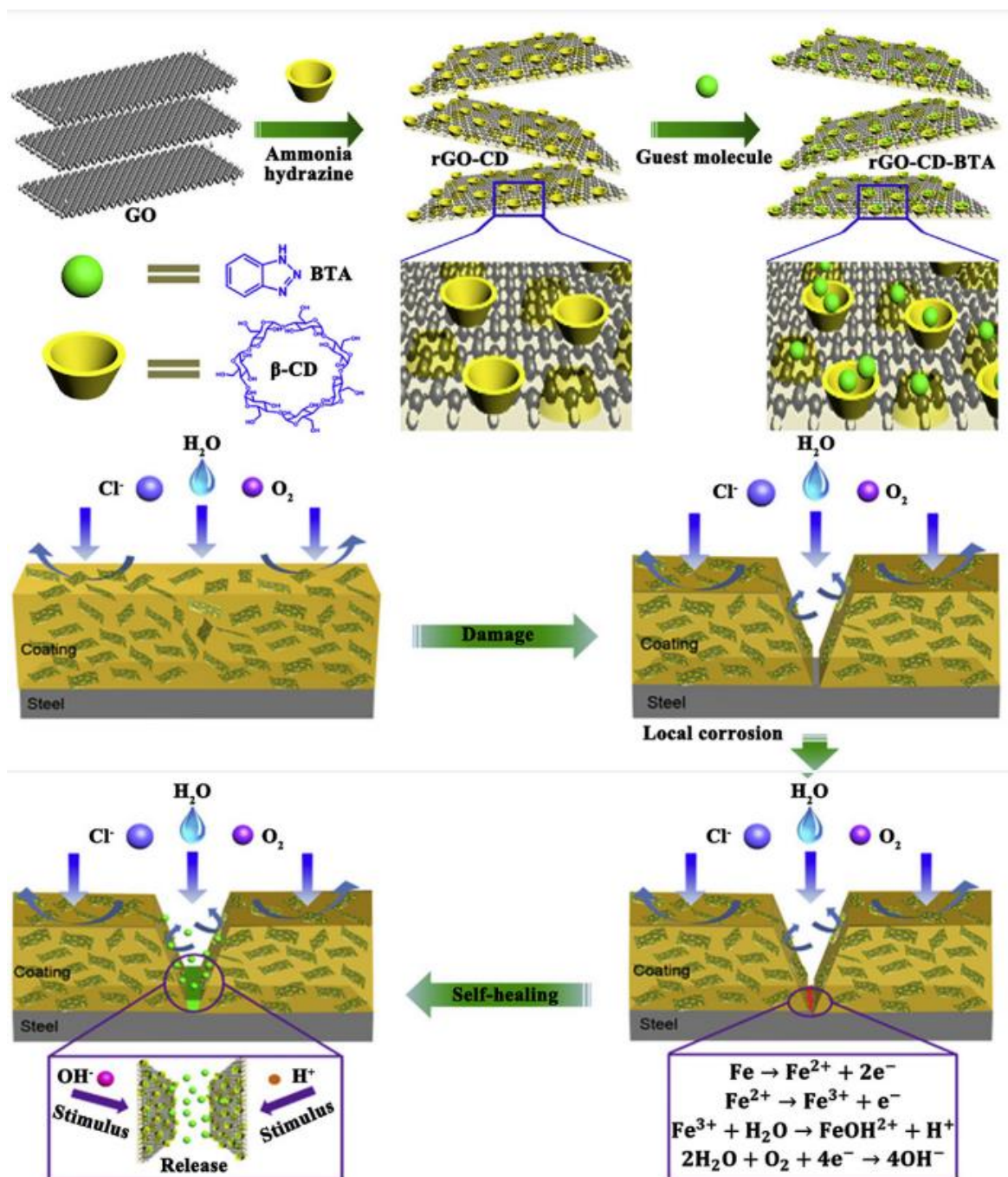


Figure 10. Illustration of the procedure for preparing graphene-cyclodextrin nanocontainers and loading the benzotriazole molecules, and schematic representation of the self-healing mechanism for graphene-based containers composite coatings [90].

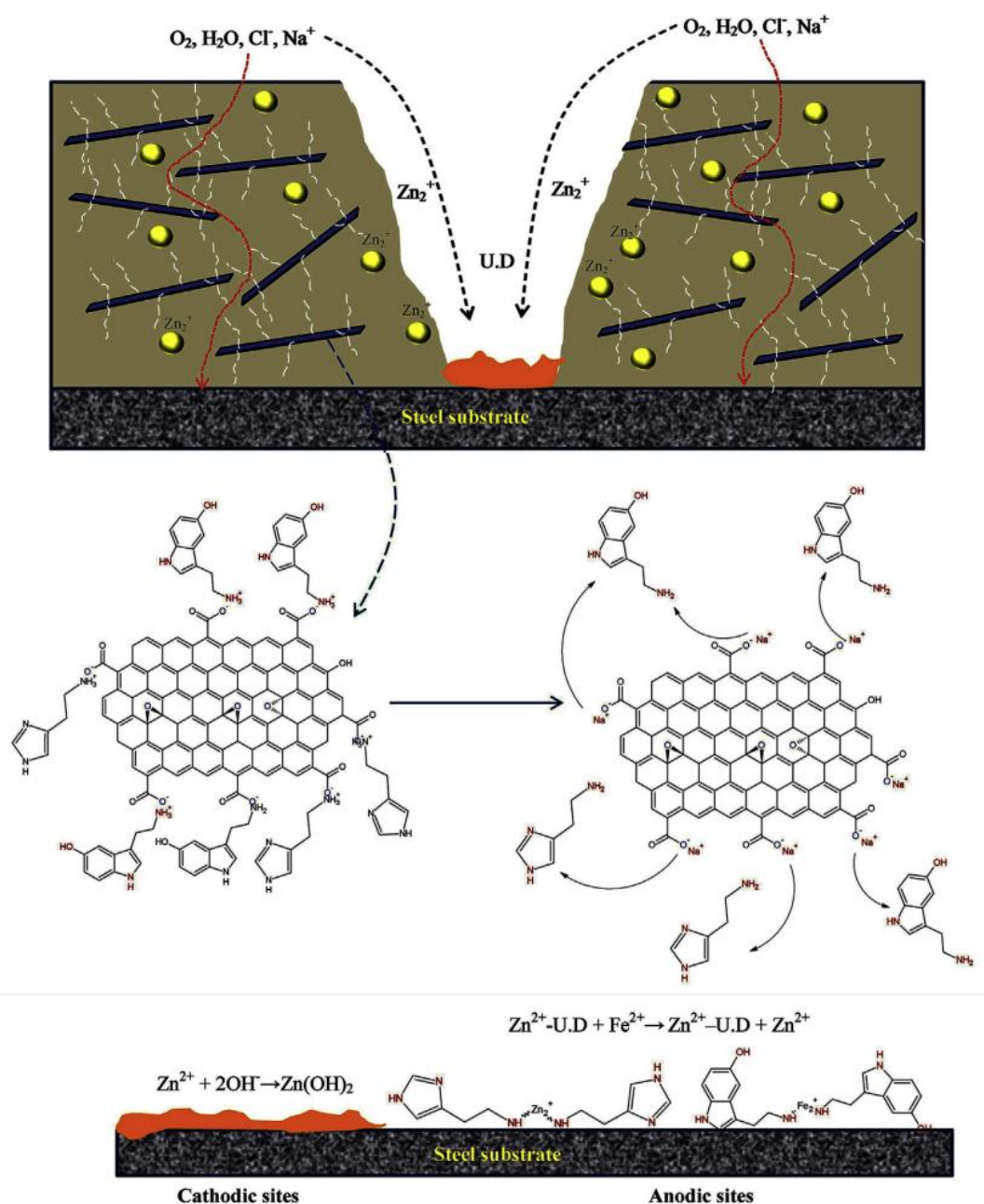


Figure 11. Mechanism of ramie leaf extract modified GO to repair defect zone of the coatings in the presence of zinc salt [91].

4. Conclusions

- GO sheets act as a corrosion inhibiting agent as protective coatings on the metals substrates in an alkaline medium. The inhibition rate of GO has been enhanced by modifying GO with various agents, as briefed out in this paper. Various factors, like a type of metals, medium, type of modifiers used to modify GO, and immersion time, *etc.*, influence the inhibition efficiency of GO.

- The highest inhibition rate was observed through the adsorption of ions Pr^{3+} , Ce^{3+} , and Zn^{2+} on the surface of GO, which was because of the exchange of these ions with Na^+ ions in the corrosion electrolyte. This may increase the self-healing ability of the inhibitor when the metals are exposed for a long period in the corrosion environment.
- Hydrophobicity attained upon modified GO coating blocks the intrusion of corrosive ions and improves inhibition efficiency owing to the adsorption of polar groups on GO onto the surface of metals.
- GO derivatives have revealed a good inhibition affinity for metals in basic solutions, and hence new derivatives have to be developed to attain maximum inhibition efficiency in acidic solutions.

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