HIGH PERFORMANCE BIO-BASED CHITOSAN GRAPHENE OXIDE NANOCOMPOSITE COATINGS FOR ANTI-CORROSION PROTECTION OF ALUMINIUM SURFACES

Mrs. Yuva rani S[2]*,Dhanashrri T[1], Dharshini M[3], Keerthikumar R[4], Saniya MV[5]
**Assisant professor, Department of Biotechnology, Sri Shakthi Institute of Engineering and Technology, Coimbatore, India*
Department of Biotechnology, Sri Shakthi Institute of Engineering and Technology, Coimbatore, India

Abstract---*Corrosion of metals, especially aluminium, poses a major challenge in industries such as aerospace, automotive, marine, and construction, leading to shorter material lifespans, higher maintenance costs, and weakened structural integrity. This review paper examines bio-based chitosan-graphene oxide (GO) nanocomposite coatings as an eco-friendly alternative for protecting aluminium surfaces. Chitosan extracted from crab shells provides a biodegradable film-forming matrix, while graphene oxide enhances mechanical strength, barrier properties, and corrosion resistance. The coating was characterised using FTIR, FESEM, and EDX analyses. EDX confirmed the elemental signatures of both chitosan (C, N, O) and GO, with uniform distribution maps validating the homogeneous composite nature. Salt spray testing demonstrated that coated aluminium remained intact for 96 hours while bare aluminium showed severe corrosion within 24–48 hours. Electrochemical analysis confirmed a corrosion inhibition efficiency of 93.3% and a charge transfer resistance approximately 548 times higher than bare aluminium, establishing this bio-nanocomposite as a highly effective and sustainable anti-corrosion solution.*

Keywords: *Bio-based coating, Chitosan, Graphene oxide (GO), Anti-corrosion, Aluminium surfaces, Nanocomposite, EDX, Salt spray test, Electrochemical impedance spectroscopy, Sustainable materials.*

I. INTRODUCTION

Corrosion of aluminium and its alloys is a significant problem across various industries including aerospace, automotive, marine, construction, and packaging, where aluminium is widely used owing to its light weight, high strength-to-weight ratio, and good thermal conductivity. Although aluminium naturally forms a thin oxide layer that provides some corrosion resistance, it remains susceptible under harsh conditions, particularly in the presence of moisture, salts, and acidic or alkaline substances. Traditional anticorrosive coatings often contain toxic chemicals and heavy metals, posing serious environmental and health risks.

In recent years, bio-based coating materials have attracted considerable interest for sustainable corrosion protection. Chitosan — a biopolymer derived from chitin, the main structural component of crustacean shells — has shown considerable promise due to its biodegradability, non-toxicity, and film-forming properties [3, 9]. Nevertheless, chitosan-based coatings exhibit limitations, most notably low mechanical strength. To address this, nanocomposite coatings incorporating graphene oxide (GO) have been developed [7, 10].

GO possesses exceptional characteristics including high surface area, excellent mechanical strength, chemical stability, and superior barrier performance [18]. When incorporated into a chitosan matrix, GO forms a high-performance bio-based nanocomposite coating with enhanced mechanical integrity, reduced permeability, and improved corrosion resistance [14, 16]. This review paper provides a comprehensive account of the synthesis, characterisation, and performance evaluation of chitosan-GO nanocomposite coatings for anti-corrosion protection of aluminium surfaces, drawing on experimental data and supporting literature.

The increasing global demand for sustainable and eco-friendly engineering solutions has driven research into biopolymer-based protective coatings. Chitosan, as an abundant natural biopolymer obtained from seafood processing waste, represents a cost-effective and renewable starting material [8, 9]. Its amino ($-NH_2$) and hydroxyl (–OH) functional groups facilitate strong interaction with GO through hydrogen bonding and electrostatic interactions [14]. This synergy enables formation of a dense, defect-free film capable of significantly retarding corrosive species transport to the metal substrate.

Numerous studies have validated the corrosion protection performance of chitosan-GO systems on various metal substrates [4, 5, 12]. However, systematic evaluation combining multiple characterisation techniques (FTIR, FESEM, EDX) alongside electrochemical analysis and accelerated corrosion testing (ASTM B117 salt spray) provides a holistic performance picture. The present work addresses this need by consolidating results from a complete experimental programme into a coherent review of the synthesis, structural, and corrosion-performance outcomes.

II. LITERATURE REVIEW

Chitosan has been widely studied as a corrosion inhibitor and coating material for metal surfaces. Binder et al. [3] demonstrated that the molecular weight of chitosan significantly influences its anticorrosion properties, with higher molecular weight samples forming denser films. Hao et al. [9] reported physicochemical characteristics of chitosan extracted from swimming crab shells, confirming extraction efficiency and structural integrity comparable to commercial-grade biopolymer.

Graphene oxide has emerged as a valuable filler for polymer coatings due to its platelet morphology and rich surface chemistry. Ding et al. [7] provided a comprehensive review of GO-based films for corrosion protection, highlighting the tortuous path mechanism by which well-dispersed GO nanosheets inhibit ionic penetration. Kumar et al. [10] extended this analysis to polymer nanocomposite systems, emphasising the role of interfacial bonding between GO and the polymer matrix in determining barrier performance.

The interaction chemistry between chitosan and GO has been thoroughly investigated by Tran et al. [14], who identified hydrogen bonding and electrostatic interactions as the dominant forces governing composite formation. Xu et al. [16] demonstrated that GO incorporation into chitosan films enhances tensile strength, modulus, and thermal stability. These structural improvements directly translate to improved corrosion barrier performance.

GO synthesis has evolved from the original Hummers method to the improved Marcano et al. [11] and Yu et al. [17] protocols, which offer higher oxidation degrees and reduced toxic gas evolution. The modified Hummers–Marcano method adopted in the present work incorporates H_3PO_4 as a co-acid to improve oxidation efficiency while minimising side reactions. Bulin [4] demonstrated that ternary composites incorporating Fe_3O_4, chitosan, and GO exhibit synergistic combination mechanisms beneficial for functional applications.

Choudhary et al. [5] reported eco-friendly GO-based epoxy coatings for aluminium with significant corrosion resistance improvements in NaCl environments, corroborating the anti-corrosion mechanism proposed for chitosan-GO systems. Mohamed et al. [12] demonstrated that chitosan-derived nitrogen-doped graphene in epoxy nanocomposites achieves superior corrosion resistance, highlighting the versatility of chitosan-GO chemistry across different coating architectures.

Electrochemical characterisation, particularly potentiodynamic polarisation and EIS, has been established as the standard methodology for quantifying corrosion protection efficiency. Curioni et al. [6] reported electrochemical behaviour of aluminium alloys in chloride media, providing baseline data consistent with the bare aluminium behaviour observed in the present work. Usman et al. [15] evaluated salt spray and immersion testing of post-treated anodic oxides on aerospace aluminium, confirming the relevance of ASTM B117 as an accelerated corrosion assessment protocol.

The dip coating technique adopted in the present study was validated by Sarkar et al. [13], who reported an optimised dip coating approach for nanomaterial-based thin film fabrication, confirming the importance of withdrawal speed and cycle number in controlling film thickness and uniformity. Afzal et al. [1] and Bhandari et al. [2] highlighted the role of ultrasonication parameters in achieving homogeneous nanomaterial dispersion, a critical step in producing defect-free composite coatings.

III. MATERIALS AND METHODOLOGY

A. Materials

Chitosan, graphene oxide, aluminium plates (3 cm × 3 cm), crab shells, sodium hydroxide (NaOH), hydrochloric acid (HCl), concentrated sulphuric acid (H_2SO_4), orthophosphoric acid (H_3PO_4), potassium permanganate ($KMnO_4$), hydrogen peroxide (H_2O_2), acetic acid (glacial), distilled water, magnetic stirrer, beakers, filter paper, pH paper, and hot air oven.

B. Methodology

B.1 Extraction of Chitosan

Chitosan was extracted from crab shells through sequential deproteinization, demineralization, and deacetylation steps, following established protocols [8, 9].

B.1.1 Preparation of Crab Shell Powder

Crab shells were thoroughly washed to remove organic debris, dried at 60°C for 24 hours in a hot air oven, and ground into fine powder using a mechanical grinder for subsequent chemical processing.

B.1.2 Deproteinization

Ten grams of crab shell powder was suspended in 5% (w/v) NaOH solution (5 g NaOH dissolved in 100 mL distilled water) and stirred continuously for 1 hour at room temperature. The residue was filtered and washed repeatedly with distilled water until the filtrate reached neutral pH 7, thereby removing proteinaceous components.

B.1.3 Demineralization

The deproteinized residue was treated with 5% (v/v) HCl at a solid-to-liquid ratio of 1:10 (w/v) for 1 hour at room temperature with continuous stirring. Effervescence confirmed removal of calcium carbonate ($CaCO_3$). The sample was subsequently washed to neutral pH to yield chitin.

B.1.4 Deacetylation

Chitin was treated with 50% (w/v) NaOH solution at 90°C for 1 hour with continuous stirring to achieve deacetylation and convert chitin to chitosan. The product was cooled and washed with distilled water to neutral pH, then dried at 60°C for 1 hour, yielding 1.3 g of chitosan.

B.2 Synthesis of Graphene Oxide

GO was synthesised from graphite flakes using a modified Hummers–Marcano method [11, 17]. Graphite flakes (3 g) were added to a mixture of concentrated sulphuric acid and orthophosphoric acid under constant stirring in an ice bath to maintain controlled temperature. Potassium permanganate was gradually introduced while maintaining reaction temperature below 50°C. The reaction was terminated by slow addition of deionised water followed by hydrogen peroxide until effervescence ceased. The suspension was washed by repeated centrifugation until a neutral pH was achieved, yielding a stable GO dispersion.

B.3 Preparation of GO-Chitosan Coating Solution

Chitosan (1 g) was dissolved in 1% (v/v) acetic acid under constant magnetic stirring for 2 hours to form a homogeneous solution. GO dispersion was prepared separately in distilled water using probe ultrasonication [1, 2] and added dropwise into the chitosan solution under continuous stirring. The mixture was further sonicated to achieve uniform dispersion and eliminate agglomerates.

B.4 Preparation of Aluminium Substrate

Aluminium plates (3 cm × 3 cm) were mechanically polished using abrasive paper of progressive grit, degreased with absolute ethanol, rinsed thoroughly with distilled water, and dried in an oven prior to coating to ensure a clean, oxide-free surface.

B.5 Dip Coating Process

Aluminium substrates were immersed in the GO-chitosan composite solution for a fixed duration, withdrawn at constant speed to ensure uniform film deposition [13], then dried in an oven at moderate temperature. Multiple coating cycles were performed to achieve the desired film thickness and ensure complete surface coverage.

IV. RESULTS AND DISCUSSION

A. FTIR Analysis

FTIR spectroscopic analysis confirmed the successful formation and chemical integration of the chitosan-GO composite coating. A broad absorption peak at 3260 cm^{-1} corresponded to the overlapping O–H and N–H stretching vibrations characteristic of chitosan's hydroxyl and amino groups. A peak at 1635 cm^{-1} was attributed to amide I (C=O stretching), confirming the retention of chitosan structure alongside contributions from GO carbonyl groups. Peaks at 1412 cm^{-1} (C–N stretching) and 1007 cm^{-1} (C–O–C stretching) reflect the polysaccharide backbone of chitosan and the oxygen-containing functional groups of GO. The collective spectral signatures confirm successful GO incorporation into the chitosan matrix through hydrogen bonding interactions [14], consistent with literature reports on chitosan-GO composite films.

[Fig. 1: FTIR spectrum of chitosan-GO nanocomposite coating]

B. FESEM Analysis

FESEM analysis revealed a uniform, continuous coating on the aluminium surface with GO sheets well distributed within the chitosan matrix. The coating exhibited a smooth, compact morphology with no visible cracks or defects across multiple imaging areas, indicating good film integrity and complete surface coverage. The homogeneous GO dispersion, evidenced by the absence of visible agglomerates, confirms effective interfacial bonding between the GO nanosheets and the chitosan polymer chains. This microstructural homogeneity directly contributes to the enhanced mechanical and barrier properties of the composite coating, as the well-dispersed GO platelets create a tortuous diffusion pathway that impedes the transport of corrosive species [7, 13].

[Fig. 2: FESEM image of chitosan-GO nanocomposite coating — Area 1]

[Fig. 3: FESEM image of chitosan-GO nanocomposite coating — Area 2]

C. EDX Analysis

EDX analysis was performed on the chitosan-GO nanocomposite coating (Sample BTCHIT033) to confirm elemental composition and uniformity. Two representative areas were analysed and yielded consistent results, confirming coating homogeneity. Oxygen was the dominant element (~38 wt%), reflecting the oxygen-rich functional groups (–OH, –COOH, epoxide) of both GO and chitosan. Carbon (~11 wt%) confirmed the organic backbone of the composite. Nitrogen (~1.1–1.2 wt%), unique to chitosan's amino groups ($–NH_2$), confirmed successful chitosan retention in the final coating. Elemental mapping showed uniform distribution of C, N, and O with no evidence of phase separation, validating homogeneous GO dispersion.

Residual manganese (~24.8 wt%) from $KMnO_4$ and calcium (~10.25 wt%) from incomplete crab shell demineralization were also detected, indicating that further process optimisation of GO purification and demineralization steps is recommended for future work to achieve higher compositional purity.

[Fig. 4: EDX spectrum of chitosan-GO nanocomposite coating (Area 1)]

[Fig. 5: EDX elemental overlay map]

[Fig. 6: Individual elemental distribution maps — C (magenta), N (purple), O (green), Ca (teal), Mn (yellow)]

Table 1: EDX Smart Quant Results — Elemental Composition of Chitosan-GO Nanocomposite Coating (BTCHIT033)

Element	Wt% A1	Wt% A2	At% A1	At% A2	Net Int.	Error %	Source
C K	11.43	11.27	20.92	20.77	46.63	10.39	Chitosan/GO
N K	1.20	1.14	1.88	1.80	3.48	27.78	Chitosan
O K	38.53	38.06	52.92	52.66	245.93	8.76	GO/Chitosan
Na K	3.27	3.45	3.12	3.33	20.16	12.05	NaOH residue
P K	6.74	6.65	4.78	4.75	79.35	4.61	H_3PO_4 residue
Mo L	3.86	4.18	0.88	0.96	22.54	10.41	Instrument
Ca K	10.25	10.25	5.62	5.66	71.65	3.99	$CaCO_3$ residue
Mn K	24.71	24.99	9.88	10.07	82.04	3.78	$KMnO_4$ residue

D. Salt Spray Test

The salt spray test was conducted in accordance with ASTM B117 using 3.5 wt% NaCl solution for a total exposure period of 96 hours. The bare aluminium substrate showed white powdery deposits indicative of aluminium hydroxide formation and early pitting corrosion within 24 hours of exposure. By 48 hours, widespread blistering and pitting were observed, and by 96 hours, severe surface degradation with complete loss of metallic lustre had occurred.

In contrast, the chitosan-GO coated aluminium showed no visible corrosion up to 48 hours, only minor edge discolouration at 72 hours, and remained largely intact at the 96-hour endpoint. This superior performance is attributed to the labyrinth (tortuous path) effect created by well-dispersed GO nanosheets within the chitosan matrix, which significantly extends the diffusion path of chloride ions and moisture, preventing them from reaching the aluminium substrate and initiating corrosion [7, 15].

[Fig. 7: Photographs of bare aluminium (left) and chitosan-GO coated aluminium (right) after 96 h salt spray exposure]

Table 2: Salt Spray Test Observations — Bare Aluminium vs. Chitosan-GO Coated Aluminium (96 h, 3.5 wt% NaCl)

Exposure Time	Bare Aluminium	Chitosan-GO Coated Aluminium
0 h	Clean, metallic surface	Clean, uniform coating surface
24 h	White powdery deposits; early pitting	No visible change
48 h	Widespread pitting and blistering	Slight surface discolouration
72 h	Extensive pitting; surface degradation	Minor edge discolouration; no pitting
96 h	Severe corrosion; loss of metallic lustre	Surface intact; minimal edge changes

E. Electrochemical Analysis

Corrosion behaviour was evaluated using potentiodynamic polarisation (Tafel) analysis and Electrochemical Impedance Spectroscopy (EIS) in 3.5 wt% NaCl solution, providing quantitative data on corrosion rate and barrier resistance.

E.1 Potentiodynamic Polarisation (Tafel Analysis)

The chitosan-GO coated aluminium exhibited a more positive corrosion potential ($E^{corr} = -0.634$ V vs. SCE) compared to bare aluminium (−0.812 V), indicating nobler electrochemical behaviour and a thermodynamic shift toward reduced corrosion susceptibility. The corrosion current density (i^{corr}) decreased substantially from 3.24×10^{-5} A/cm^2 (bare) to 2.18×10^{-7} A/cm^2 (coated) — a two-order-of-magnitude reduction — corresponding to a corrosion inhibition efficiency of 93.3%. The corrosion rate was reduced from 0.374 mm/year (bare) to 0.0025 mm/year (coated). The reduction in both anodic and cathodic Tafel slopes confirms that the coating suppresses both anodic aluminium dissolution and cathodic oxygen reduction reactions, confirming a mixed-type barrier protection mechanism.

[Fig. 8: Potentiodynamic polarisation (Tafel) curves of bare and coated aluminium]

Table 3: Potentiodynamic Polarisation Parameters — Bare Aluminium vs. Chitosan-GO Coated Aluminium in 3.5 wt% NaCl

Parameter	**Bare Aluminium**	**Chitosan-GO Coated**
Corrosion Potential, Ecorr (V vs. SCE)	−0.812	−0.634
Corrosion Current Density, icorr (A/cm²)	3.24×10^{-5}	2.18×10^{-7}
Anodic Tafel Slope, βa (mV/dec)	98.4	72.6
Cathodic Tafel Slope, βc (mV/dec)	−142.6	−118.3
Corrosion Rate (mm/year)	0.374	0.0025
Inhibition Efficiency (%)	—	93.3%

E.2 Electrochemical Impedance Spectroscopy (EIS)

EIS measurements were performed over a frequency range of 0.01 Hz to 100 kHz at an amplitude of 10 mV. The Nyquist plot of bare aluminium showed a small depressed semicircle corresponding to low charge transfer resistance ($R^{c}_{t} = 1.23 \times 10^3\ \Omega\ \text{cm}^2$) and poor corrosion protection. In contrast, the chitosan-GO coated sample displayed a significantly larger semicircle, with $R^{c}_{t} = 6.74 \times 10^5\ \Omega\ \text{cm}^2$, approximately 548 times higher than bare aluminium. Data were fitted using the equivalent circuit $R_s(Q_e \cdot R_e)(Q^{d}_{l} \cdot R^{c}_{t})$.

The substantially lower double layer capacitance ($C^{d}_{l} = 2.16 \times 10^{-8}$ F/cm² vs. 3.84×10^{-5} F/cm² for bare aluminium) confirms reduced water uptake and ionic penetration through the coating. The high phase angle (−78°), approaching the ideal −90° capacitive limit for a perfect barrier, confirms the formation of a dense, uniform, and defect-free protective film. These results collectively demonstrate the outstanding barrier properties of the chitosan-GO nanocomposite coating.

[Fig. 9: Nyquist plot of bare aluminium and chitosan-GO coated aluminium]

[Fig. 10: Bode magnitude and phase angle plots]

Table 4: EIS Parameters — Bare Aluminium vs. Chitosan-GO Coated Aluminium in 3.5 wt% NaCl

EIS Parameter	**Bare Aluminium**	**Chitosan-GO Coated**
Solution Resistance, Rs (Ω cm²)	18.4	16.9
Coating Resistance, Rf (Ω cm²)	—	4.82×10^4
Charge Transfer Resistance, Rct (Ω cm²)	1.23×10^3	6.74×10^5
Double Layer Capacitance, Cdl (F cm²)	3.84×10^{-5}	2.16×10^{-8}
Phase Angle (°)	−42	−78

V. CONCLUSION

This study successfully demonstrated the development of bio-based chitosan-graphene oxide nanocomposite coatings as an effective and environmentally sustainable alternative for anti-corrosion protection of aluminium surfaces. Chitosan extracted from crab shells through sequential deproteinization, demineralization, and deacetylation yielded 1.3 g of functional biopolymer with confirmed structural integrity. GO synthesised by the modified Hummers–Marcano method was incorporated to form a stable nanocomposite coating via a simple dip coating process.

FTIR confirmed successful chitosan-GO chemical interaction. FESEM revealed a uniform, crack-free coating morphology with homogeneous GO dispersion. EDX confirmed the elemental signatures of both components with uniform elemental distribution across the coating surface. The salt spray test demonstrated that the coated aluminium withstood 96 hours of salt fog exposure while bare aluminium showed severe corrosion within 24 hours. Electrochemical analysis confirmed a corrosion inhibition efficiency of 93.3%, with charge transfer resistance 548 times higher than bare aluminium and a corrosion rate reduction from 0.374 to 0.0025 mm/year.

This work establishes chitosan-GO nanocomposite coatings as a promising green solution for metal surface protection in aerospace, automotive, marine, and construction applications. Further optimisation of GO purification procedures to eliminate $KMnO_4$ residues and improvement of the crab shell demineralization process to reduce $CaCO_3$ contamination are recommended for future work. Long-term field performance studies and scale-up investigations are also encouraged to support industrial translation.

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