

Sustainable Epoxy Nanocomposite Coating from Eggshell-Derived CaO Nanoparticles and *Phyllanthus amarus* Extract for Corrosion Protection of Tinplate

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Abstract

A sustainable epoxy nanocomposite coating incorporating eggshell-derived calcium oxide nanoparticles (CaONPs) and aqueous *Phyllanthus amarus* leaf extract was developed for corrosion protection of tinplate. CaONPs were prepared through calcination–hydration–recalcination of waste eggshells and characterized using UV–Vis, FTIR, XRD, SEM and EDX techniques which confirmed the formation of a crystalline cubic CaO nanoparticles. Phytochemical screening of the extract revealed tannins, phenolics, flavonoids, alkaloids and glycosides, supporting its potential as a plant-derived corrosion inhibitor. The anticorrosion performance of epoxy formulations containing CaONPs (NP), plant extract (PE), their nanocomposite (NC), and no additive was evaluated by immersion in 5 wt% NaCl and accelerated salt-spray testing. NC exhibited the lowest immersion corrosion rate ($\approx 0.035\text{--}0.038\text{ mm y}^{-1}$) and maintained approximately 68–71% inhibition efficiency. Following salt-spray exposure, NC showed only $1.2 \pm 0.5\text{ mm}$ rust creep and $2.0 \pm 1.0\%$ corroded area, representing reductions of approximately 90.8 and 97.3%, respectively, relative to the no-additive coating. Kinetic analysis further demonstrated markedly suppressed corrosion propagation. The enhanced performance is attributed to a complementary barrier–inhibition effect involving CaONP-induced reinforcement of the epoxy matrix and phytochemical-assisted interfacial inhibition, demonstrating a promising waste-derived and bio-based strategy for tinplate corrosion protection.

Keywords: Calcium oxide nanoparticles; *Phyllanthus amarus*; Epoxy nanocomposite coating; Corrosion inhibition; Tinplate.

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1. Introduction

Corrosion is an inevitable interfacial degradation process that affects metallic materials exposed to chemically or electrochemically aggressive environments. Its consequences extend beyond material loss to include deterioration of structural integrity, shortened service life, increased maintenance requirements, contamination of products, and substantial economic losses. The global economic burden associated with corrosion has historically been estimated at several percent of gross domestic product, making effective corrosion control important across manufacturing, transportation, infrastructure, energy, and packaging industries. Accordingly, protective coatings and corrosion inhibitors remain among the most practical strategies for restricting interactions between metallic surfaces and aggressive species such as water, dissolved oxygen, chloride ions, and acidic constituents (Bhat et al., 2024; Holla et al., 2024; Sheydaei, 2024).

Tinplate, consisting essentially of a thin steel sheet protected by an electrodeposited tin layer, is particularly important in the packaging industry because it combines the mechanical strength and formability of steel with the appearance and corrosion resistance of tin (Pandey et al., 2023). Despite these advantages, the tin coating does not provide absolute protection. Surface defects, pores, mechanical damage, or progressive deterioration of the protective layer may permit aggressive species to reach the underlying steel. Chloride-containing environments are particularly relevant because chloride ions can contribute to localized attack and deterioration of metallic surfaces. Recent investigations of commercial tinplate have demonstrated that corrosion behaviour is strongly influenced by environmental factors including NaCl concentration and temperature (Hussein et al., 2023). Consequently, improving the barrier and inhibitive properties of coatings applied to tinplate remains important for prolonging material service life.

Organic polymer coatings provide a convenient means of separating a metallic substrate from its surrounding environment. Among these materials, epoxy resins are widely employed because of their strong adhesion, chemical resistance, mechanical properties, ease of application, and ability to form relatively dense barrier films (Randis et al., 2023; Zaghoul et al., 2023). However, conventional epoxy coatings are not completely impermeable. Micropores, microcracks and other defects may arise during curing or service, providing pathways through which water, oxygen and aggressive ions can migrate towards the coating–metal interface. Prolonged exposure can therefore progressively diminish the protective performance of the coating (Zaghoul et al., 2023). One increasingly important approach for overcoming these limitations is the incorporation of nanoscale inorganic fillers into polymer matrices. Properly dispersed nanoparticles can increase the tortuosity of diffusion pathways, reduce coating porosity and improve interfacial, mechanical and barrier properties (Liu et al., 2024; Randis et al., 2023).

Metal-oxide nanoparticles are particularly attractive as functional additives for polymeric anticorrosion coatings. Their small dimensions, relatively high surface area and physicochemical properties can modify coating microstructure and impede the movement of corrosive species. Recent assessments of nanoadditives have shown that nanomaterials can improve the corrosion resistance, strength and durability of polymer coatings, while combinations of functional additives may produce synergistic protective effects (Liu et al., 2024). Calcium oxide nanoparticles (CaONPs) represent an interesting candidate in this context because CaO can be produced from inexpensive calcium-rich biological waste. Chicken eggshells, which consist predominantly of calcium carbonate, provide an abundant precursor that can be thermally transformed to CaO. Recent studies have demonstrated the successful preparation of crystalline CaO nanoparticles from waste eggshells through calcination, providing a route for converting an otherwise discarded material into a value-added nanomaterial (Adaikalam et al., 2024; Hemmami et al., 2024).

The use of eggshell waste as a precursor for CaONPs also introduces a circular-economy dimension into coating development. Rather than relying entirely on virgin inorganic precursors, waste eggshells can be washed, processed and calcined to convert CaCO_3 to CaO, thereby simultaneously addressing waste valorisation and functional-material production. For example, a recent study demonstrated an environmentally favourable conversion of waste eggshells to CaO nanoparticles by calcination at 900 °C (Adaikalam et al., 2024). Furthermore, a similar study reported eggshell-derived CaO nanoparticles with well-defined structural and morphological characteristics (Hemmami et al., 2024). These studies demonstrate the potential of eggshells as low-cost feedstocks for nanomaterial synthesis and support their further exploration in environmentally oriented applications.

In parallel with nanomaterial modification, plant-derived substances have received increasing attention as alternatives to conventional corrosion inhibitors. Some traditional inhibitors provide high protection efficiencies but may present toxicity, persistence or disposal concerns. Plant extracts offer a potentially more sustainable

approach because they are renewable and contain naturally occurring organic compounds capable of interacting with metallic surfaces (Holla et al., 2024; Izuagie et al., 2023; Li et al., 2025; Sheydaei, 2024).

Their inhibitive behaviour is commonly associated with phytochemicals such as phenolics, flavonoids, tannins, alkaloids and related compounds containing heteroatoms, aromatic systems and functional groups capable of interacting with metal surfaces. Adsorption of these constituents can reduce the number of electrochemically active sites and establish a protective interfacial film that limits anodic and/or cathodic corrosion reactions (Li et al., 2025; Sheydaei, 2024).

Phyllanthus amarus (*P. amarus*) is particularly relevant in this regard. Previous studies have established that extracts of *P. amarus* can inhibit metallic corrosion through adsorption of their organic constituents on the substrate. Okafor et al. (2008) demonstrated the inhibitory action of *P. amarus* extracts on mild steel in acidic environments, while subsequent electrochemical investigations attributed its performance to adsorption of organic constituents at the metal–solution interface (Anupama et al., 2016; Okafor et al., 2008). Although these earlier studies were conducted primarily by introducing the extract directly into corrosive media, they provide an important basis for investigating *P. amarus* as a functional component incorporated into protective coating systems. The phytochemical screening conducted in the present work identified tannins, phenolics, flavonoids, alkaloids and glycosides in the plant extract, further supporting its potential contribution to interfacial corrosion inhibition.

Combining plant-derived inhibitors with inorganic nanoparticles in an epoxy matrix may therefore provide complementary modes of protection. The epoxy phase can function primarily as a physical barrier, whereas dispersed nanoparticles may increase diffusion-path tortuosity and improve coating integrity. At locations where electrolyte penetration occurs, adsorbing phytochemical constituents may provide an additional inhibitive contribution at the metal/coating interface. Such a multifunctional strategy is consistent with current efforts to develop greener anticorrosion coatings incorporating bio-derived compounds and nanomaterials rather than relying exclusively on conventional synthetic inhibitors (Bhat et al., 2024; Izuagie et al., 2025; Li et al., 2025; Zaghloul et al., 2023). Recent literature has consequently emphasized hybrid and nanocomposite systems as promising routes towards durable, multifunctional and environmentally responsible corrosion protection (Deyab et al., 2024; Liu et al., 2024; Randis et al., 2023).

Despite considerable research on plant extracts as corrosion inhibitors and the rapid development of nanoparticle-modified epoxy coatings, comparatively less attention has been directed towards integrating waste-derived CaO nanoparticles and *P. amarus* extract within a single epoxy coating for tinplate protection in chloride media. This approach provides an opportunity to combine waste valorisation, renewable phytochemicals, nanoscale barrier enhancement and conventional polymer-coating technology in one protective system. The present study therefore develops an epoxy-based nanocomposite coating containing CaONPs prepared from waste chicken eggshells and *P. amarus* leaf extract and evaluates its ability to protect tinplate against corrosion. The prepared CaONPs were characterized using UV–visible spectroscopy, Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and scanning electron microscopy (SEM), while corrosion protection was assessed under chloride exposure using immersion/weight-loss and accelerated salt-spray approaches. The work thus explores a potentially low-cost and sustainable route for developing multifunctional anticorrosive coatings from locally accessible plant material and biogenic waste.

2. Materials and Methods

2.1 Materials and Instrumentations

Analytical-grade reagents were used throughout the study. Waste chicken eggshells, used as the precursor for calcium oxide nanoparticles (CaONPs), were collected from local tea sellers within Sokoto metropolis, Nigeria. Fresh leaves of *P. amarus* (stonebreaker) were collected from the vicinity of Sokoto State University and identified with voucher number UDUH/ANS/0497. The coating matrix consisted of a two-component epoxy resin system comprising resin and hardener mixed in an 80:20 proportion (PPG Industries). Sodium chloride (NaCl, 99.5%) and distilled water were used to prepare the corrosive medium. Other chemicals used included sodium hydroxide (NaOH, 99.0%), hydrochloric acid (HCl, 36 %), n-hexane (99.0%), all supplied by Qualikems India, JHD China and Sigma Aldrich. Major equipment used in this study included a hot-air oven (Gallenkamp, UK), furnace capable of operation at 900 °C (Narange Medical Ltd., India), analytical balance (OHAUS SPU-202, USA), a fabricated salt-spray accelerated corrosion test chamber (see **Figure 1**), paint plastic mixing cups (LAKGRUPPEN, Denmark), tinplate test panels (Pro Test Panels Ltd., England), and a Dino-Lite digital microscope (AM2011, Scitek Global Co., Ltd., China). Ultraviolet-visible (UV-Vis) analysis was carried out on a UV-1800 Shimadzu UV spectrophotometer using standard cuvettes (d = 10.00 mm) with a resolution of 1 nm between 250 and 800 nm. Fourier transform infrared spectroscopy (FTIR) analyses were conducted on a Bruker Alpha spectrometer fitted with a Platinum ATR module (4000 – 500 cm⁻¹). Spectra were recorded for solid samples after drying for at least 1 h. Scanning electron microscopy (SEM, Phonem Prox, Malaysia) coupled with Energy dispersive X-ray (EDX), was operated at an accelerated voltage of 10 kV. XRD analysis was conducted using XRD, Philips X' Pert Pro with Cu K α radiation of wavelength of 0.15416 nm at the range of 5 - 80°.

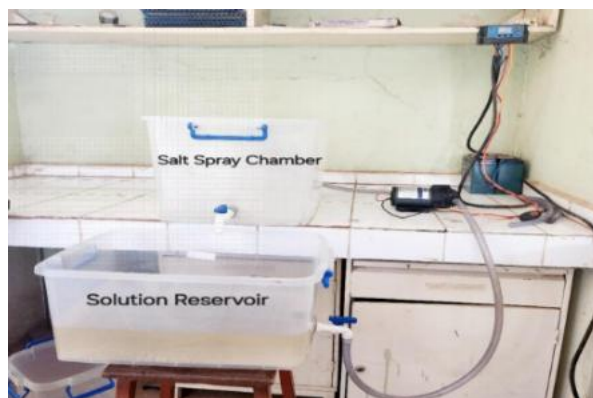


Figure 1. Fabricated laboratory salt-spray accelerated corrosion testing setup, comprising an upper salt-spray chamber and a lower saline-solution reservoir connected by a pumping system for generating a continuous salt mist to evaluate coating durability under simulated saline conditions.

2.2 Pretreatment, extraction and screening of *P. amarus* leaves

Fresh *P. amarus* leaves were thoroughly washed with clean water to remove adhering dust and other surface contaminants. The cleaned leaves were shade-dried under laboratory conditions for ten days and subsequently pulverized using a mortar and pestle. The resulting material was sieved to obtain a uniform fine powder. For aqueous extraction, 20 g of the powdered plant material was soaked in 100 ml of boiling distilled water and allowed to extract for 3 h with intermittent agitation. The mixture was subsequently filtered to separate the plant residue from the aqueous extract. The resulting filtrate was concentrated and freeze-dried to obtain the crude aqueous extract (Onwuliri et al., 2009). The dried extract was transferred into airtight containers and stored at 4 °C until required for phytochemical screening and preparation of the nanocomposite coating.

2.3 Collection and Pretreatment of Eggshells

Waste chicken eggshells were washed with warm water to remove adhering organic matter, rinsed thoroughly with distilled water, and dried in a hot-air oven at 100 °C for 24 h. The dried shells were crushed using a blender and sieved to obtain a fine eggshell powder for the preparation of CaONPs.

2.4 Phytochemical Screening of *P. amarus* Extract

Qualitative phytochemical screening was carried out to determine the major classes of secondary metabolites present in the *P. amarus* extract, including tannins, alkaloids, saponins, flavonoids, glycosides, phenolics, steroids, and terpenoids. For tannins, approximately 200 mg of extract was boiled with 10 mL of distilled water and treated with 0.1% ferric chloride; development of a blue-black colour was taken as a positive result. Alkaloids were tested by acidifying an aqueous portion of the extract with 1% HCl and treating it with Mayer-Wagner reagent; formation of a cream or brown-red precipitate indicated a positive test. Saponins were assessed by vigorous shaking of the extract with distilled water until observance of persistent frothing. Flavonoids were tested by treating an ethanolic filtrate of the extract with concentrated HCl and magnesium ribbon, with pink or red coloration indicating their presence. Glycosides were assessed by treating the extract with distilled water and NaOH and observing the development of a yellowish colour (Bilgic, 2022; Ogunleye et al., 2020).

2.5 Preparation of Calcium Oxide Nanoparticles from Eggshells

CaONPs were prepared by adapting standard methods (Adaikalam et al., 2024; Putkham et al., 2022). The powdered eggshells were calcined in a furnace at 900 °C for 3 h to decompose the calcium carbonate (CaCO_3) component to calcium oxide (CaO). The resulting CaO was refluxed in water at 60 °C for 6 h, during which hydration produced calcium hydroxide, Ca(OH)_2 . The product was dried in a hot-air oven at 105 °C overnight, ground to a fine powder, and calcined again at 900 °C for 3 h to dehydrate Ca(OH)_2 and obtain highly active CaONPs. The final product was stored under vacuum in a desiccator until use.

2.6 Characterization of CaONPs

UV-Vis spectroscopy was used to examine the optical absorption behaviour of the prepared CaONPs over the ultraviolet-visible region. FTIR spectra were recorded in the mid-infrared region (approximately 4000–400 cm^{-1}) to identify characteristic functional groups and bonding environments associated with the nanoparticles. XRD analysis was performed using Cu $K\alpha$ radiation over an appropriate 2θ range to evaluate the crystalline phases and structure of the product. Surface morphology and particle aggregation were examined by SEM after suitable sample mounting and preparation.

2.7 Formulation and Application of the Nanocomposite Coating

The nanocomposite coating was formulated using CaONPs, *P. amarus* extract, and a two-component epoxy system. The epoxy resin and hardener were mixed in a plastic mixing cup at a mass ratio of 4:1 (80:20, w/w). CaONPs and *P. amarus* extract were incorporated into the epoxy system at an approximately 1:1 mass ratio (0.98 and 0.95 g, respectively) and mechanically mixed for 10 min to obtain a homogeneous dispersion. The resulting nanocomposite formulation was applied uniformly to degreased tinplate panels (50 × 25 × 1 mm) using a film applicator. The coated specimens were then allowed to cure at ambient temperature for 24 h before corrosion testing. Similar approaches involving the incorporation of nanoparticles and plant-derived extracts into epoxy matrices have previously been reported (Mod et al., 2024).

2.8 Corrosion Resistance Evaluation

Corrosion performance was assessed using gravimetric immersion tests in 5 wt% NaCl and accelerated salt-spray testing (ASTM B117-23), combined with surface examination to evaluate corrosion resistance and coating durability under simulated saline conditions.

2.8.1 Immersion Test

For the immersion test, coated tinplate specimens were immersed in sodium chloride solution for predetermined exposure periods at room temperature for 90 days. At each sampling interval, the specimens were removed from the solution, cleaned, dried, and weighed. The change in mass was used to determine weight loss and to assess the corrosion performance of the coating. Visual examination was also carried out to document rusting, pitting, blistering, and other evidence of coating failure. Corrosion rate (CR) was calculated by:

$$CR = \frac{87.6 \times W}{D \times A \times T}$$

where W = weight loss (mg), D = density (g cm^{-3}), A = area (cm^2), T = time (h) (Fontana, 2005). The inhibition efficiency (η %) was derived as:

$$\eta = \frac{CR_0 - CR_i}{CR_0} \times 100$$

where CR_0 = corrosion rate without inhibitor and CR_i = rate with inhibitor (Ovari et al., 2023).

2.8.2 Accelerated Salt-Spray Test and Surface Examination

Corrosion performance of the coated and X-scribed panels was evaluated according to ASTM B117-23 (ASTM International, 2019). Samples were exposed to a continuous 5 wt % NaCl fog at 35 °C and 95 % relative humidity in a salt-spray chamber. At scheduled intervals (24, 48, 72, 96, 168, 240 and 360 h), panels were withdrawn, rinsed with deionized water, and air-dried for image analysis. Surface degradation was documented using a digital optical microscope (10×–200×, reflected bright-field illumination) or an SEM following ASTM D1654 procedures (ASTM International, 2024). Micrographs of the scribed regions were captured at fixed magnification and lighting to record rust propagation, blistering, and coating breakdown. Image analysis provided rust-creep length and corroded-area fraction, while time-to-first-rust and extent of film deterioration were noted visually. Prolonged resistance to rust initiation and lower rust-creep or corroded-area values were taken as indicators of superior coating protection (ASTM International, 2024).

2.9 Surface Degradation and Corrosion-Kinetic Analysis

The extent of surface deterioration was evaluated from the progression of corrosion on the exposed specimens. Where rust-creep length and corroded-area fraction were determined from surface images, lower values were interpreted as indicating better coating protection. Time-dependent corrosion data were further assessed using the parabolic and power-law kinetic models. The goodness of fit was evaluated using the coefficient of determination (R^2), while the fitted rate constants and power-law exponent were used to compare the progression of corrosion among the coating systems.

2.10 Data Analysis

Experimental measurements were summarized using appropriate descriptive statistics in Microsoft Excel. Where replicate measurements were available, results were expressed as mean $\pm$ standard deviation. Regression analysis was used for kinetic-model fitting, and R^2 values were used to assess the agreement between the experimental data and the fitted models.

3. Results and Discussion

3.1 Phytochemical screening

As shown in **Table 1**, qualitative phytochemical screening of the aqueous *P. amarus* leaf extract revealed the presence of tannins, phenolics, flavonoids, alkaloids, and glycosides, whereas saponins, terpenoids, and steroids were not detected. The detected constituents are broadly consistent with previous reports demonstrating that *P. amarus* contains several classes of bioactive phytochemicals, although their occurrence varies with extraction conditions and solvent polarity (Asaduzzaman et al., 2026; Berezi et al., 2020). These phytochemicals contain functional groups capable of interacting with metal surfaces and promoting protective-film formation. This supports the potential use of *P. amarus* as a green corrosion inhibitor, consistent with previous studies demonstrating its effective adsorption and corrosion inhibition on metallic substrates (Okafor et al., 2008).

Table 1. Qualitative Phytochemical Constituents of the Aqueous *P. amarus* Leaf Extract

Phytoconstituents	Inference
Saponin	–
Tannin	+
Phenolic	+
Flavonoid	+
Alkaloid	+
Glycoside	+
Terpenoid	–
Steroid	–

Note: (+) = detected; (–) = not detected.

3.2 Characterization of CaONPs

3.2.1 Percentage Yield and Physical Appearance of CaONPs

The synthesis produced a 47.3 % yield of CaONPs, obtained as a white powder (**Table 2**). The white appearance is consistent with the characteristic physical appearance of calcium oxide and indicates substantial removal of the organic components initially associated with the eggshell precursor during high-temperature treatment. The moderate yield can be attributed largely to mass loss accompanying thermal decomposition of the predominantly CaCO_3 eggshell matrix, during which CO_2 was evolved to form CaO. Recent studies continue to demonstrate the feasibility of converting eggshell waste into CaO nanomaterials by thermal and related synthesis routes, providing a useful pathway for valorizing calcium-rich biowaste (Thabet et al., 2026).

Table 2. Percentage yield and colour of CaONPs

Sample	% Yield	Colour
CaONPs	47.3 %	White

3.2.2 UV–Visible Spectroscopic Analysis

The UV–Vis spectrum of the synthesized CaONPs (**Figure 2**) exhibits a pronounced absorption band centred at approximately 300 nm, with a maximum absorbance of about 0.80, followed by a sharp decrease in absorption above approximately 350–400 nm. This strong absorption in the UV region is consistent with the optical behaviour expected for wide-band-gap calcium oxide materials. UV absorption of CaO nanoparticles has been

associated with electronic transitions involving the oxide valence band and calcium-derived conduction states, although the exact absorption maximum can vary considerably with synthesis route, particle size, defects and surface chemistry. Recent studies have similarly employed UV–Vis spectroscopy alongside XRD and FTIR to confirm the optical characteristics of eggshell-derived CaO nanomaterials (Inbakumar et al., 2025). The relatively low absorption in the visible region indicates that the synthesized material interacts predominantly with ultraviolet rather than visible radiation.

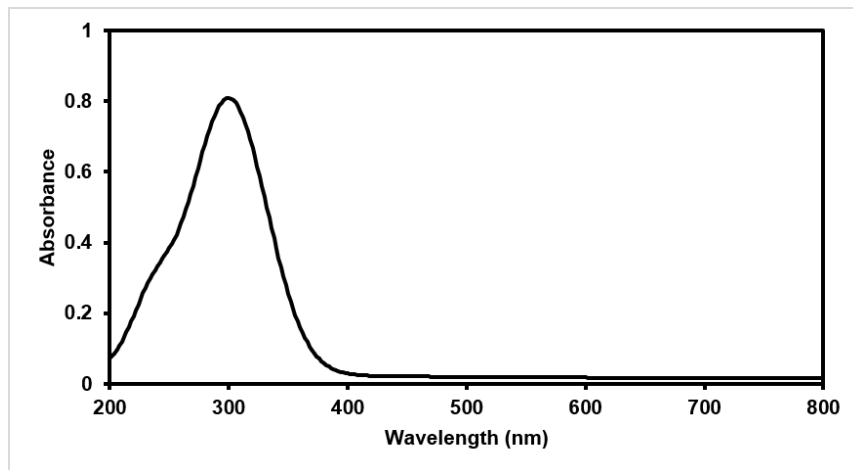


Figure 2. UV–Vis absorption spectrum of the synthesized CaONPs.

3.2.3 FTIR Analysis

The FTIR spectrum (**Figure 3**) provides information on the chemical bonding and surface species associated with the synthesized nanoparticles. A sharp absorption feature at approximately 3642 cm^{-1} can be assigned to O–H stretching, which may arise from surface hydroxyl groups or partial hydration of CaO to $\text{Ca}(\text{OH})_2$. CaO is highly hygroscopic and readily interacts with atmospheric moisture during sample handling. A similar band near 3644 cm^{-1} has been reported for CaO-containing materials and attributed to adsorbed moisture/hydroxyl species (Mostafa et al., 2023).

The band around 1416 cm^{-1} is consistent with carbonate-related C–O vibrations. Its presence suggests limited surface carbonation resulting from interaction of the highly reactive CaO surface with atmospheric CO_2 . Likewise, the feature at approximately 914 cm^{-1} may be associated with carbonate/hydroxyl-related vibrations. Recent work on eggshell-derived CaO has similarly reported residual carbonate bands, emphasizing that CaO readily undergoes re-carbonation and hydration following calcination (Inbakumar et al., 2025). Most importantly, the pronounced absorption toward the low-wavenumber region, particularly below approximately 600 cm^{-1} , is consistent with metal–oxygen vibrations associated with Ca–O bonding. Ca–O absorption around $455\text{--}512\text{ cm}^{-1}$ has been reported for calcium oxide materials (Mostafa et al., 2023). Overall, the FTIR spectrum supports CaO formation while indicating some degree of surface hydroxylation and carbonation, which is expected for this highly reactive oxide.

3.2.4 X-ray Diffraction Analysis

The XRD pattern (**Figure 4**) displays several sharp and well-defined diffraction peaks, demonstrating the crystalline nature of the synthesized material. The principal reflections occur at approximately 32° , 37° , 54° , 64° and 67° (2θ) and are indexed in the supplied diffractogram as the (111), (200), (220), (311) and (222) planes, respectively. The intense peak near 37° indicates a strongly crystalline phase. These positions are broadly consistent with the characteristic reflections of cubic CaO, for which major diffraction peaks are commonly observed around 32° , 37° , 54° , 64° and 67° (2θ). Recent studies of CaO nanoparticles synthesized from eggshells have likewise used XRD to demonstrate formation of a crystalline cubic CaO phase (Rekha et al., 2024).

The sharpness and relatively narrow widths of the reflections indicate good crystallinity. Importantly, the XRD pattern provides considerably stronger evidence for CaO formation than UV–Vis spectroscopy alone. In conjunction with the Ca–O-related FTIR absorption, the diffraction pattern therefore supports the successful conversion of the calcium-rich eggshell precursor into crystalline CaO.

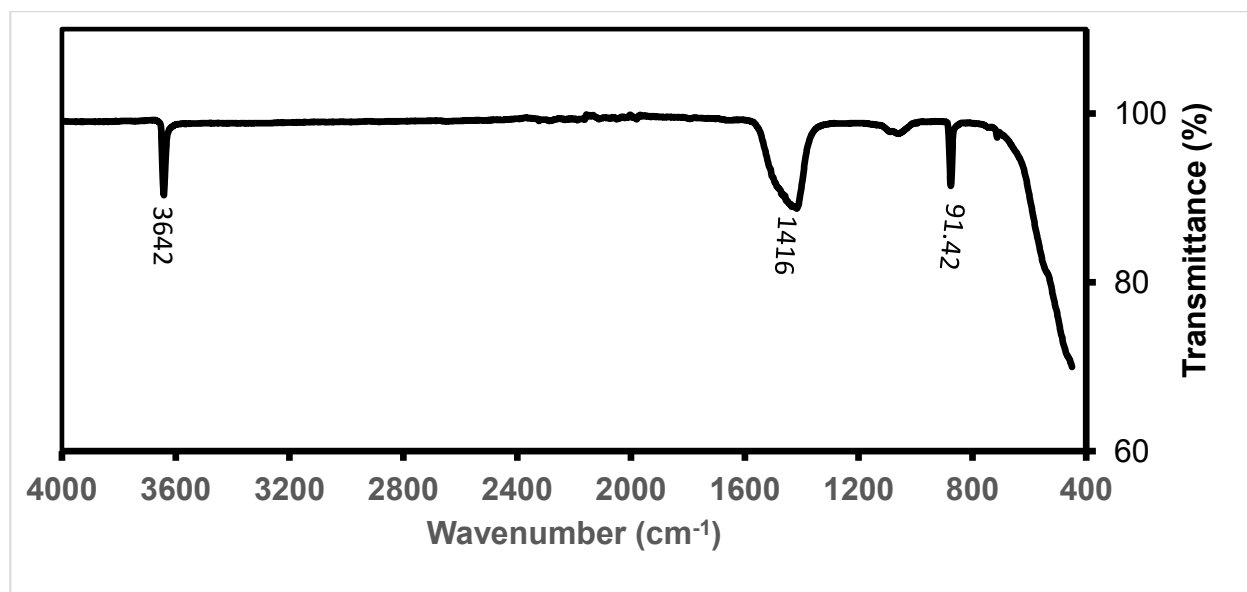


Figure 3. FTIR spectrum of the synthesized CaONPs.

3.2.5 SEM-EDX Morphological Analysis

The SEM micrograph obtained at 100,000× magnification with a 200 nm scale bar (**Figure 5**) reveals numerous small, approximately spherical particles assembled into larger irregular aggregates. The individual primary particles appear nanoscale, while pronounced agglomeration produces interconnected clusters. Such agglomeration is common in CaO nanopowders and may arise from the high surface energy of nanoscale particles, which promotes particle–particle interactions during calcination, drying and storage. Recent characterization of eggshell-derived CaO nanomaterials has similarly reported spherical or irregular nanoscale particles with aggregation, while the exact morphology and dimensions vary with synthesis conditions. For example, a recent study on eggshell-derived CaO reported predominantly spherical nanoparticles and emphasized the influence of processing conditions on morphology (Inbakumar et al., 2025).

The morphology observed here may be advantageous for incorporation into an epoxy coating because the fine particulate structure can potentially occupy microvoids within the polymer matrix and increase the tortuosity of diffusion pathways. Nevertheless, effective dispersion during coating preparation is important because excessive agglomeration can instead create coating heterogeneity and defects. Thus, the SEM result supports the formation of a finely divided CaO material suitable for subsequent evaluation as a nanofiller in the developed anticorrosion coating.

Equally, the SEM–EDX analysis (**Figure 6**) confirms that the synthesized nanoparticles are predominantly composed of calcium (Ca) and oxygen (O). The EDX spectrum shows strong O K α and Ca K α /K β signals, with quantitative analysis giving 71.23 wt% Ca and 28.77 wt% O, corresponding to atomic percentages of 47.45 for Ca and 52.55 for O. The atomic composition is therefore close to the expected 1:1 Ca:O stoichiometry of CaO. Elemental mapping further shows that Ca and O are broadly distributed throughout the particle aggregates, supporting formation of a relatively homogeneous CaO material. The absence of detectable additional elements in the presented spectrum also suggests low levels of elemental impurities within the analysed region. Similar Ca/O compositions have been reported for eggshell-derived CaONPs, including 73.27 wt% Ca and 26.73 wt% O

in a recent study (Hemmami et al., 2024). These EDX results, when considered together with the XRD and FTIR results, provide complementary evidence supporting the successful formation of CaONPs.

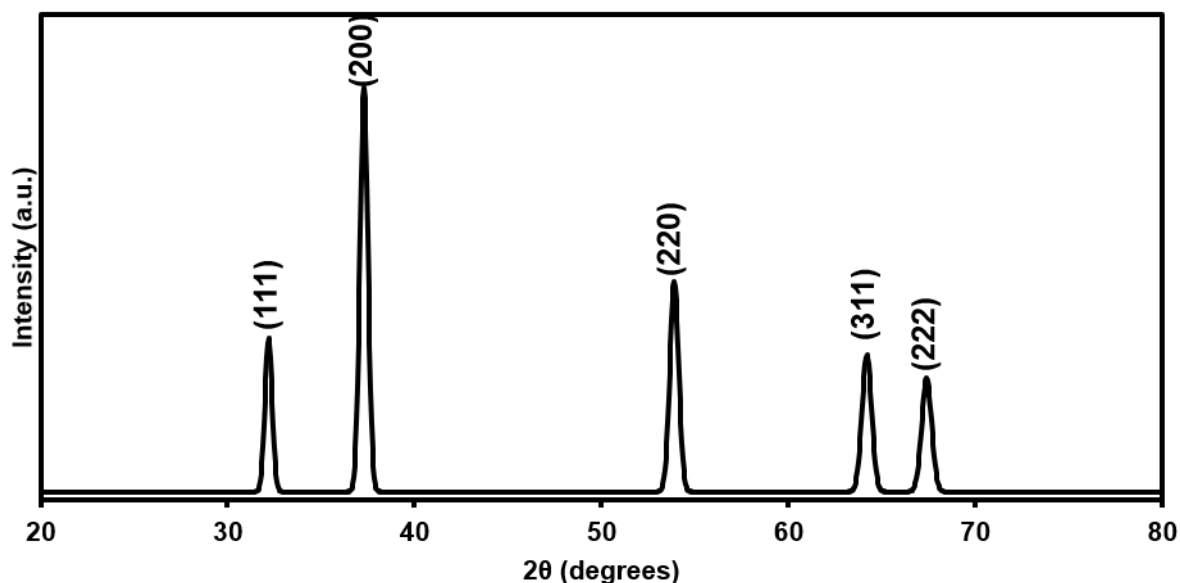


Figure 4. XRD pattern of the synthesized CaONPs showing the characteristic crystalline diffraction peaks.

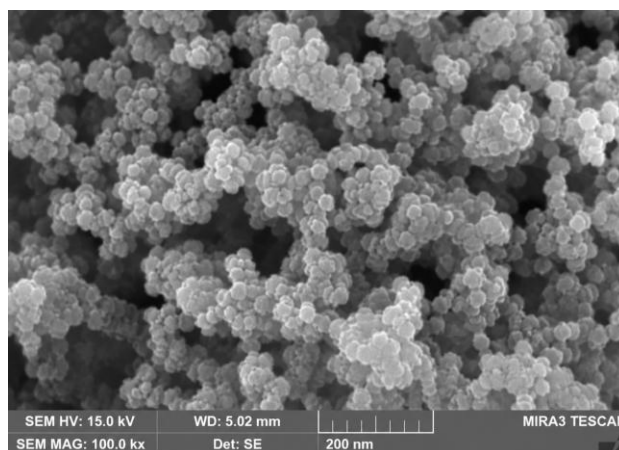


Figure 5. SEM micrograph of the synthesized CaONPs showing agglomerated, approximately spherical particles at 100,000× magnification.

3.3 Anticorrosion Studies

3.3.1 Immersion corrosion behaviour

The immersion results demonstrate that incorporation of CaONPs and *P. amarus* extract markedly improved the corrosion resistance of epoxy-coated tinplate. As shown in **Figure 7**, the corrosion rate increased gradually with exposure time, following the order No Additive > NP > PE > NC. The no-additive coating increased from approximately 0.109 to 0.130 mm y⁻¹, whereas the nanocomposite (NC) maintained the lowest values (≈0.035–0.038 mm y⁻¹), indicating sustained protection during exposure.

The improved performance of NP is consistent with the earlier characterization of the CaONPs. UV–Vis, FTIR and XRD analyses supported CaO formation, while SEM revealed nanoscale, approximately spherical particles and EDX showed Ca and O as the dominant elements. When incorporated into epoxy, such inorganic

nanoparticles can act as fillers that restrict electrolyte penetration and increase the tortuosity of diffusion pathways through coating defects. Nanoparticle modification is consequently an established strategy for enhancing polymer-coating barrier properties.

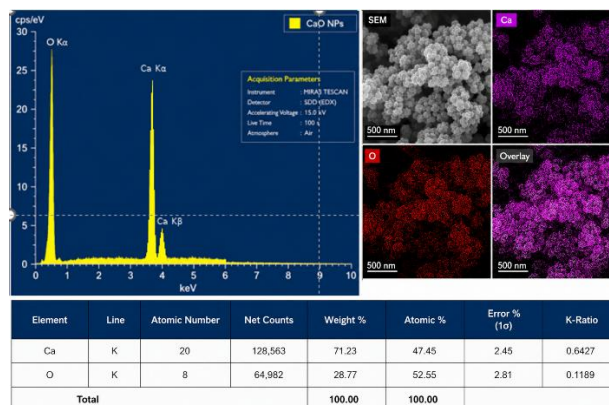


Figure 6. EDX spectrum and elemental mapping of the synthesized CaONPs, showing the distribution and elemental composition of calcium (Ca) and oxygen (O).

PE performed better than NP alone, which may be attributed to the phytochemicals identified in the *P. amarus* extract, particularly tannins, phenolics, flavonoids, alkaloids and glycosides. Plant-derived inhibitors containing heteroatoms, aromatic systems and π -electrons can adsorb at metal surfaces and form protective layers that restrict electrochemical reactions (Barbu et al., 2025; Sheydaei, 2024).

The superior NC performance therefore suggests a complementary barrier-inhibition effect, combining the physical barrier contribution of CaONPs with phytochemical-assisted interfacial inhibition. This interpretation is supported by **Figure 8**, where NC maintained approximately 68–71% inhibition efficiency, compared with 44–45% for PE and 20–22% for NP. The relatively stable efficiencies with exposure time further indicate persistent protection by the hybrid coating.

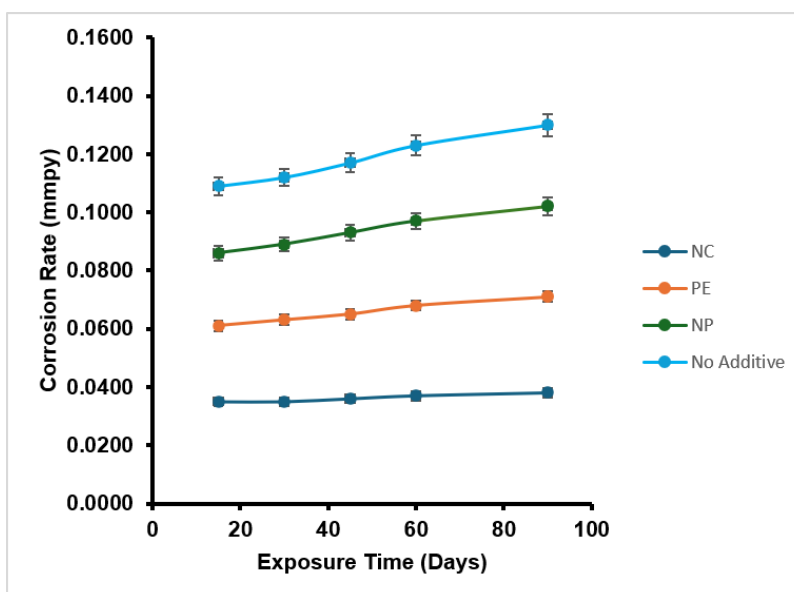


Figure 7. Variation of corrosion rate with exposure time for tinplate specimens coated with nanocomposite (NC), *P. amarus* extract (PE), CaONPs (NP), and epoxy without additive during corrosion exposure. Error bars represent the standard deviation of replicate measurements.

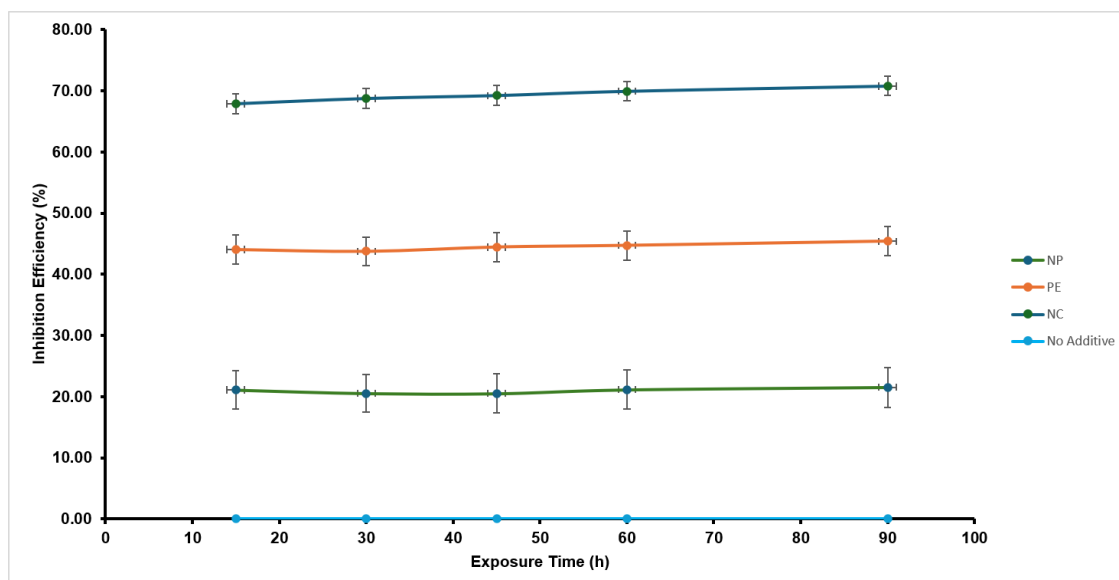


Figure 8. Variation of corrosion inhibition efficiency with exposure time for tinplate specimens coated with CaONPs (NP), *P. amarus* extract (PE), CaONP/*P. amarus* nanocomposite (NC), and epoxy without additive. Error bars represent the standard deviation of replicate measurements.

3.3.2 Accelerated salt-spray performance and surface morphology

The accelerated salt-spray results corroborate the immersion tests and demonstrate the superior protection afforded by the nanocomposite coating. SEM images of the scribed surfaces (**Figure 9**) reveal extensive deterioration and corrosion-product accumulation around the defect in the no-additive coating. Damage decreased progressively for NP and PE, whereas the NC surface remained comparatively intact, with corrosion largely confined to the scribe.

The quantitative results (**Table 3**) support these observations. The no-additive coating exhibited a rust-creep length of 13.0 ± 2.0 mm and $75.0 \pm 5.0\%$ corroded area. These decreased to 7.0 ± 1.5 mm and $40.0 \pm 5.0\%$ for NP and 4.0 ± 1.0 mm and $25.0 \pm 5.0\%$ for PE. NC provided the greatest protection, with only 1.2 ± 0.5 mm rust creep and $2.0 \pm 1.0\%$ corroded area, corresponding to reductions of approximately 90.8% and 97.3%, respectively, relative to the no-additive coating.

Since the scribe directly exposes the substrate to the corrosive environment, the limited propagation observed for NC indicates effective suppression of under-film corrosion. Similar improvements have been reported for plant-extract-containing epoxy coatings, where phytochemicals provide active inhibition at damaged metal/coating interfaces (Paul et al., 2024; Ulaeto et al., 2023). This performance is also consistent with the earlier SEM characterization, which confirmed the presence of nanoscale CaO particles. When uniformly dispersed within the coating matrix, such nanofillers can enhance barrier properties by increasing the tortuosity of diffusion pathways and thereby restricting the transport of corrosive species to the metal surface (Samardžija et al., 2023). Thus, the NC performance is consistent with complementary contributions from the CaONP barrier effect and phytochemical-assisted corrosion inhibition, as similarly reported for organic-inorganic green hybrid epoxy systems (Karattu et al., 2023).

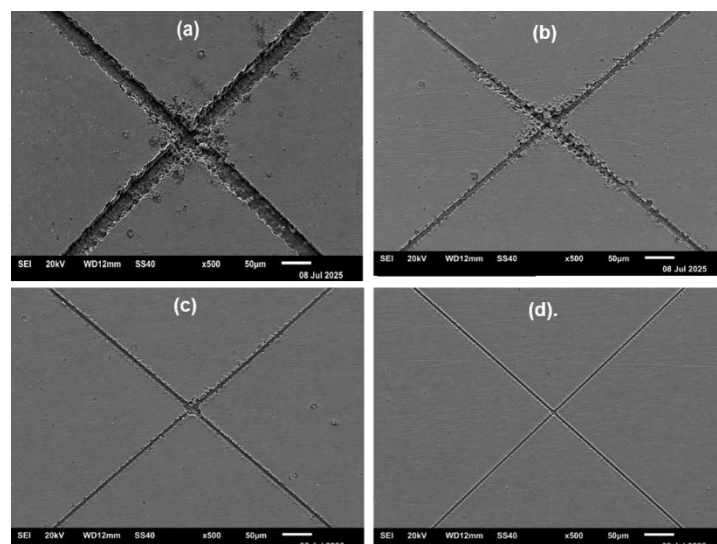


Figure 9. SEM micrographs of scribed epoxy-coated tinplate specimens after corrosion exposure: (a). coating without additive (No additive), (b). CaONP-containing coating (NP), (c). *P. amarus* extract-containing coating (PE), and (d). CaONP/*P. amarus* extract nanocomposite coating (NC).

Table 3. Rust-creep length, corroded-area fraction, and qualitative corrosion assessment of the different epoxy coating formulations after corrosion exposure.

Coating Type	Rust-Creep Length (mm)	Corroded-Area Fraction (%)	Interpretation
No Additive	13.0 ± 2.0	75.0 ± 5.0	Very severe corrosion
Nanoparticles (NP)	7.0 ± 1.5	40.0 ± 5.0	Severe to moderate corrosion
Plant Extract (PE)	4.0 ± 1.0	25.0 ± 5.0	Moderate corrosion; improved protection
Nanocomposite (NC)	1.2 ± 0.5	2.0 ± 1.0	Excellent corrosion inhibition

3.3.3 Surface degradation and corrosion kinetics

The rust-creep data in **Figure 10** and **Figure 11** further demonstrate the durability of the coating formulations. After 360 h, L^2 reached approximately 169 mm² for the no-additive coating, compared with 49 and 16 mm² for NP and PE, respectively, while NC maintained negligible propagation throughout exposure. This progressive separation indicates that the protective advantage of NC became increasingly pronounced with prolonged exposure.

The kinetic parameters (**Table 4**) support this trend. The parabolic rate constant decreased from 0.750 mm h^{-1/2} for the no-additive coating to 0.389, 0.226 and 0.066 mm h^{-1/2} for NP, PE and NC, respectively, representing an approximately 91% reduction for NC. Corresponding R^2 values of 0.841–0.965 indicate that the parabolic model describes the modified coatings reasonably well, although the poorer fit for the no-additive system suggests more heterogeneous degradation.

The power-law model, $L^2 = kt^n$, provided stronger overall fits, with R^2 values of 0.983–1.000. The rate coefficient decreased markedly from 0.437 (no additive) to 0.230 (NP), 0.209 (PE) and only 0.0032 for NC, confirming substantial suppression of corrosion propagation. Although NC exhibited $n \approx 1.049$, compared with 0.556–0.611 for the other coatings, this exponent should be considered together with its exceptionally small k and minimal observed rust creep rather than interpreted independently.

These findings agree with contemporary studies showing that well-dispersed nanofillers can reduce coating defects and extend diffusion pathways for water and aggressive ions, thereby retarding corrosion propagation (Tang et al., 2026). The combined kinetic and morphological results therefore support a barrier-inhibition mechanism, consistent with the superior performance of the CaONP/*P. amarus* hybrid coating.

Overall, the results demonstrated that the superior anticorrosion performance of NC arises from a complementary barrier-inhibition effect, combining CaONP-induced reinforcement of the epoxy matrix with phytochemical-assisted interfacial inhibition by *P. amarus*, resulting in sustained resistance to electrolyte penetration and corrosion propagation.

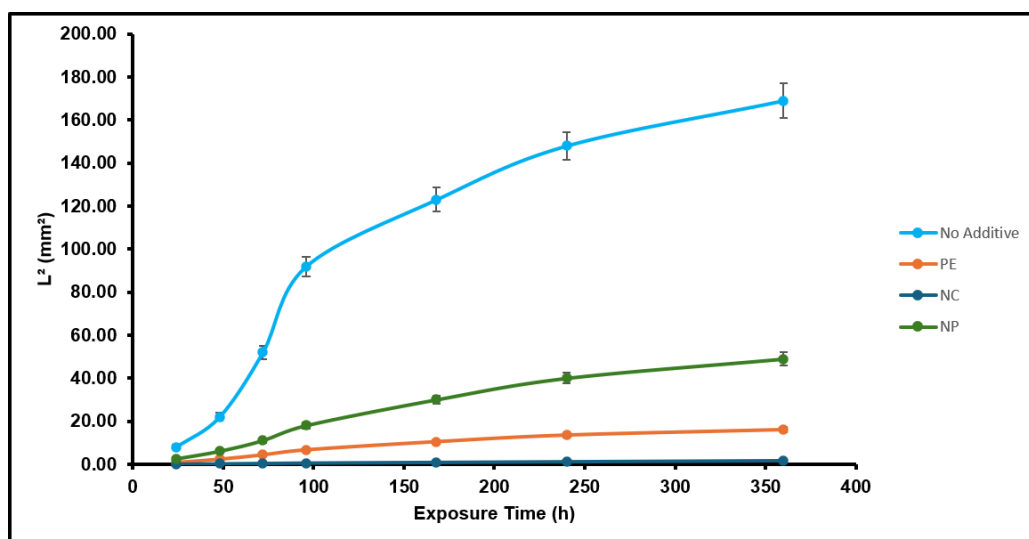


Figure 10. Parabolic rate-law plot showing the variation of rust-creep area (L^2) with salt-spray exposure time for tinplate specimens coated with CaONPs (NP), *P. amarus* extract (PE), CaONP/*P. amarus* nanocomposite (NC), and epoxy without additive. Error bars represent the standard deviation of replicate measurements.

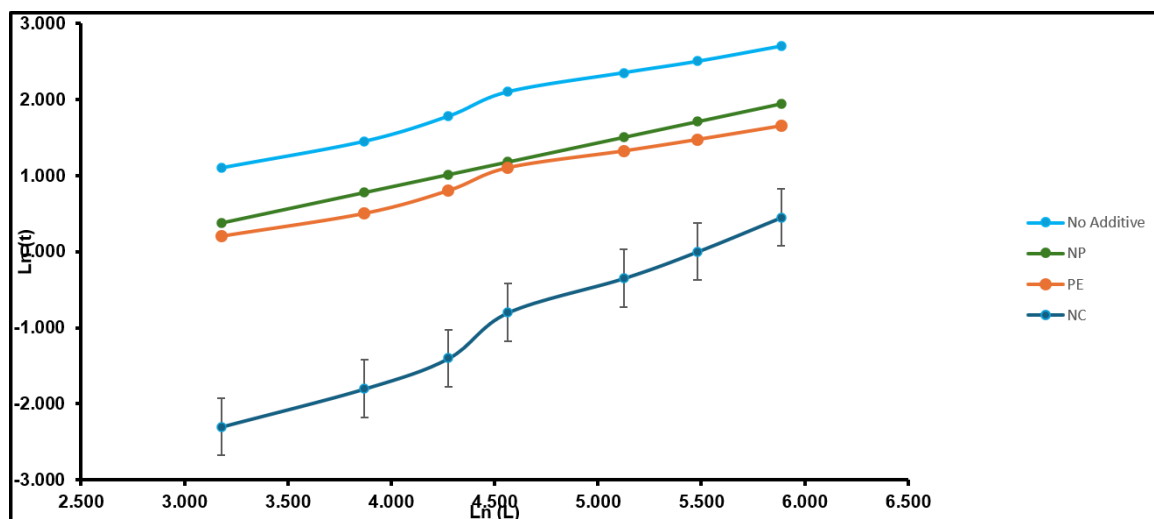


Figure 11. Power-law kinetic plot of rust-creep propagation, expressed as $\log(L^2)$ versus $\log(t)$, for tinplate specimens coated with CaONPs (NP), *P. amarus* extract (PE), CaONP/*P. amarus* nanocomposite (NC), and epoxy without additive. Error bars represent the standard deviation of replicate measurements.

Table 4. Kinetic parameters obtained from parabolic and power-law models for corrosion propagation of tinplate specimens coated with CaONPs (NP), *P. amarus* extract (PE), CaONP/*P. amarus* nanocomposite (NC), and epoxy without additive during salt-spray exposure.

Sample	Model	Rate Constant	Exponent (n)	R ²
No Additive	Parabolic	$k_p = 0.750 \text{ mm h}^{-1/2}$	–	0.841
NP	Parabolic	$k_p = 0.389 \text{ mm h}^{-1/2}$	–	0.958
PE	Parabolic	$k_p = 0.226 \text{ mm h}^{-1/2}$	–	0.921
NC	Parabolic	$k_p = 0.066 \text{ mm h}^{-1/2}$	–	0.965
No Additive	Power Law	$k = e^{-0.828} \approx 0.437$	0.611	0.983
NP	Power Law	$k = e^{-1.468} \approx 0.230$	0.5800	1.0000
PE	Power Law	$k = e^{-1.567} \approx 0.209$	0.556	0.983
NC	Power Law	$k = e^{-5.737} \approx 0.0032$	1.049	0.988

4. Conclusion

This study demonstrated the potential of a sustainable epoxy nanocomposite coating incorporating eggshell-derived CaONPs and *P. amarus* extract for protecting tinplate against corrosion in chloride environments. Characterization confirmed the formation of crystalline CaO nanoparticles, while phytochemical screening revealed tannins, phenolics, flavonoids, alkaloids and glycosides with potential corrosion-inhibiting functionalities. The combined nanocomposite (NC) formulation consistently outperformed the CaONP (NP), plant-extract (PE) and no-additive coatings. During immersion testing, NC maintained the lowest corrosion rate ($\approx 0.035\text{--}0.038 \text{ mm y}^{-1}$) and an inhibition efficiency of approximately 68–71%. Salt-spray testing further demonstrated its superior durability, with only $1.2 \pm 0.5 \text{ mm}$ rust creep and $2.0 \pm 1.0\%$ corroded area, corresponding to reductions of approximately 90.8% and 97.3%, respectively, relative to the no-additive coating. Kinetic analysis likewise showed substantially reduced corrosion propagation, with NC exhibiting the lowest parabolic and power-law rate coefficients. Overall, the findings support a complementary barrier–inhibition mechanism in which CaONPs enhance the physical barrier properties of the epoxy matrix while *P. amarus* phytochemicals contribute interfacial corrosion inhibition. The developed coating therefore provides a promising approach that combines waste-eggshell valorization, renewable plant-derived inhibitors and nanocomposite technology for improved tinplate corrosion protection.

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Conflict of Interest

The author declares no conflict of interest.

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