

Bayesian-Guided Development and Experimental Validation of a Hybrid Self-Healing Nanoencapsulated Epoxy Coating for Reinforcing Steel Applications

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ABSTRACT

This study combines formulation selection, experimental validation based on the standards, uncertainty analysis, reinforcing-bar bond, and green-cost analysis of a hybrid self-healing nanoencapsulated epoxy system with the aid of a Bayesian approach. Four coated systems (E1 – E4 consisting of 5 independent coated coupons per formulation) were compared to an uncoated reference system (B0). E4 contained graphene-oxide diffusion-barrier reinforcement, IPDI-type microcapsules, pH-responsive ZIF-8@Ca-type microfillers, MCM-41/PEI/PSS nanocontainers, and a dynamic hydrogen-bonding component. The confirmatory tests included electrochemical impedance spectroscopy tests for intact, damaged, and healed conditions; polarization-based corrosion assessment; 720 h neutral salt-fog exposure; pull-off adhesion; microcrack closure; 90-day storage-preservation assessment; and SEM/TGA characterization. Each group had a number of test specimens per coating/size group ($n = 3$, for each group), and were tested against galvanized reinforcing bars in accordance with ASTM A944. E4 produced $\log_{10}|Z|_{0.01\text{Hz}} = 11.59 \pm 0.08$ and $i_{\text{corr}} = 0.065 \pm 0.004 \mu\text{A cm}^{-2}$, corresponding to a calculated corrosion rate of $7.54 \times 10^{-4} \text{ mm y}^{-1}$ and a 97.6% reduction relative to conventional epoxy E1. E4 showed underfilm corrosion $\leq 0.35 \pm 0.04 \text{ mm}$ after 720 h of salt fog, whereas it still had $95 \pm 2\%$ adhesion and $93 \pm 3\%$ microcrack closure after 24 h. Its DQI was 0.91 (with a 0.88-0.94 uncertainty interval), and it was ranked 1st in 94% of $\pm 20\%$ weight-perturbation runs, and 91% of the 10,000 Monte Carlo draws. Two-way ANOVA for the reinforcing bar dataset showed that there was a significant difference in failure load for bar size ($p = 0.0015$) but not for surface condition (E4 vs. galvanized) ($p = 0.169$). The central plant scenario resulted in a net value of USD 32 t⁻¹ and an annual projected gain of USD 5.568 million for 174,000 t y⁻¹ of protected product. The results from all sources indicate that E4 is the recommended formulation and offer a traceable pathway for making experimental to industrial decisions.

Keywords: self-healing epoxy; nanoencapsulation; electrochemical impedance spectroscopy; Bayesian optimization; corrosion; reinforcing steel; durability-quality index; green-cost assessment

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1. INTRODUCTION

The strength of steel products is related to both the metallurgical quality and the state of the exposed surface during storage, transportation, manufacturing and use. The accumulation of scratches, pores, cut edges and handling damage in humid and chloride containing coastal and oil-field environments increases the opportunity for the electrolytes to reach the steel/coating interface and consequently reduces the durability of the coating. Once underfilm corrosion has occurred, it then spreads laterally, which leads to further cleaning, rework, product downgrade, claims, replacement requests, and the emissions of replacement steel [1].

The barrier properties, chemical resistance, adhesion, and the industrial processing of the epoxy coatings are still used today, making them the most popular material. They are most susceptible to defects, defect sensitivity, which causes a direct pathway for water, oxygen and aggressive ions if a defect is present in the polymeric barrier. Smart and self-healing coatings solve this form of damage by releasing polymerizable agents when the surface cracks, delivering inhibitors in a stimulus-responsive manner, covalently (or supramolecularly) repairing the cracks, and increasing diffusion tortuosity by the use of high-aspect-ratio fillers [2]–[4], [11], [14], [15], [18], [32], [33].

The new materials research has shifted from a single-additive modification towards a multi-mechanism one. In that, formulation discovery is found to be efficient based on machine learning for exploring the interplay of coating variables [5] and for multifunctional nanocomposites, complementary barrier and active-protection functions are exhibited for ZIF based pH responsive systems [6, 7, 10, 12, 13].

EIS is an extremely sensitive technique for monitoring water absorption, pore resistance, charge transfer at interfaces and recovery after damage in these systems [8, 9]. Damage tolerance is particularly critical for reinforcing steel as barrage bars are subjected to abrasion, bending, stacking, lifting and field handling. But an intact-film test alone isn't enough to make a high-durability system choice. The present study investigates and compares the experimental conditions of intact, mechanically damaged, and healed specimens using electrochemical testing and accelerated salt-fog testing, adhesion retention, microcrack closure, storage-preservation indicators, structural bond compatibility, and cost and environmental impact.

The lack of a single experimental-to-decision framework to connect formulation optimisation, standards-based corrosion testing, uncertainty analysis, reinforcing-bar compatibility and green-cost accounting in the plant is the main research gap. The small-data Bayesian optimization is ideal for this problem since Gaussian-process surrogates measure the uncertainty of their predictions and focus new experiments on the most informative regions of a bounded materials-design space [5] and [30]. Therefore, this study was divided into three integrated objectives: (i) identification of a hybrid self-healing epoxy formulation using Bayesian-guided screening; (ii) validation of the corrosion, adhesion, damage-recovery and product-preservation performance of the identified formulation against conventional and single-mechanism reference systems; and (iii) translation of the measured technical response into a transparent green-cost and avoided-emission decision-making model for selective industrial adoption. The study design maintains a clear separation between measured endpoints, engineering derived quantities, composite decision indices and outputs for each scenario for the plant scale.



2. SCIENTIFIC BASIS, RESEARCH GAP, AND FORMULATION LOGIC

The function of an organic coating on steel is to inhibit the passage of electrolyte, oxygen and aggressive ions to the metal/coating boundary. The barrier enhancement is therefore regulated by the film continuity, crosslink density, dispersion of the filler, water uptake and diffusion path length. Lamellar graphene oxide reinforcement makes the transport tortuous while the microcapsules provide an extrinsic repair mechanism in which the propagating crack ruptures the capsule releasing the reactive core [2], [3], [16], [17], [32].

Nanocontainer systems offer an active electrochemical function which is complimentary to mechanical sealing. The inhibitors can be released by pH-responsive ZIF-based reservoirs and mesoporous silica carriers, thereby inhibiting anodic and/or cathodic reactions in the damaged area [10, 33, 12-15, 18] when the local interfacial chemistry changes. Thus, the E4 architecture was designed by focusing on functional complementarity, in which graphene oxide was used to reinforce the barrier, microcapsules containing IPDI were used to seal cracks, MCM-41/PEI/PSS nanocontainers with ZIF-8 microfillers were used for active protection, and a dynamic hydrogen-bonding component was used to replace the local network.

The design space is clearly non-linear. Threshold behavior and interaction effects can arise from dry-film thickness, graphene-oxide fraction, loading of microcapsules, nanocontainer fraction, dynamic-bond content, curing ratio and dispersion quality. Bayesian optimization is more efficient than sequential one factor at a time experimentation of these interactions due to the use of Gaussian-process surrogate that considers response prediction with uncertainty and the use of acquisition function that considers experimenter's balance between exploration and exploitation [5] [30]. The environmental dimension is directly linked to the durability. The demand for steel production and associated carbon emissions are increased with corrosion induced replacement/rework [1]. The intensities of steelmaking are route-specific and differ markedly; according to the World Steel Association, the scrap-EAF, DRI-EAF, and BF-BOF routes have different intensities [31]. Therefore, the present green-cost framework is based on a separation of financial value from avoided-material and route-sensitive emissions accounting, for accounting traceability and to avoid double counting.

3. MATERIALS AND METHODS

The investigation included a stage of formulation-exploration and a stage of independent confirmatory comparisons. Carbon/reinforcing steel coupons were manufactured that were representative of the surface; they were identified by grade/heat, dimensions, initial mass, surface condition, coating batch, and specimen code. The surface preparation was done near-white blast cleaning (Sa 2½) according to ISO 8501-1, having a surface profile of 40–70 µm [29]. During formulation screening, the dry-film thickness (DFT) of the formulations ranged from 70 to 120 µm, and for the confirmatory comparison of E1–E4, the dry-film thickness was fixed at 90 ± 5 µm. Five independent coated coupons were used for each confirmatory formulation group. The following data were recorded to provide traceability: specimen identifiers, coating order, test order, salt-fog rack position, DFT, cure time, environmental conditions, scratch dimensions and protocol deviations. The experimental states are picked up in Table 1.

Table 1. Experimental surface-engineering states evaluated in the study.

Code	Surface state	Functional role	Use in analysis
B0	Uncoated carbon steel	No engineered barrier or	Baseline deterioration

		active healing	reference
E1	Conventional epoxy, $90 \pm 5 \mu\text{m}$	Passive barrier against water, oxygen, and chloride ingress	Industrial reference coating
E2	Epoxy + 0.05 wt% graphene oxide	Increased diffusion tortuosity	Barrier-enhanced reference
E3	Epoxy + 10 wt% IPDI-type microcapsules	Crack-triggered local polymeric sealing	Damage-healing reference
E4	Hybrid self-healing nanoencapsulated epoxy	Barrier + capsule sealing + responsive inhibitor release + dynamic bonding	Integrated high-durability system

The confirmatory E4 formulation contained 0.10 wt% graphene oxide, 10 wt% IPDI-type microcapsules, 5 wt% ZIF-8@Ca-type microfillers, 2 wt% MCM-41/PEI/PSS nanocontainers, 7 mol% dynamic-bonding component, and a curing ratio of 1.00 of stoichiometric at $90 \pm 5 \mu\text{m}$ DFT. Figure 1 summarizes the functional architecture.

Hybrid self-healing nanoencapsulated coating architecture

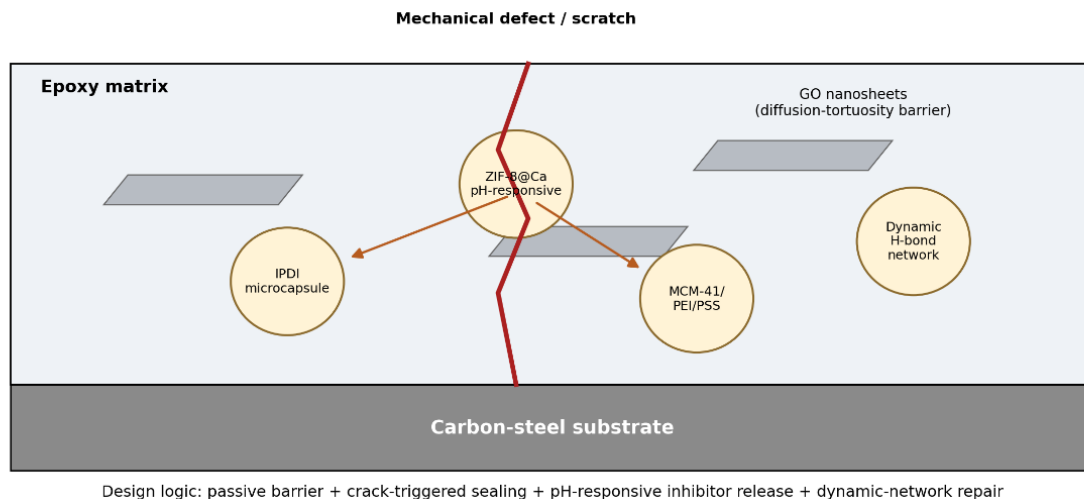


Figure 1. Hybrid self-healing nanoencapsulated E4 architecture: passive barrier reinforcement, crack-triggered sealing, pH-responsive active protection, and dynamic-network repair.

The formulation search started with 20 Latin-hypercube formulations taken throughout the bounded design space. The GP surrogate used a Matérn 5/2 kernel and automatic relevance determination, which was updated after every experimental batch. The acquisition function used was "expected improvement". As many as 30 sequential candidates were tested after the initial design, stopping when there was a marginal DQI improvement, when the budget was completed, or when the technical constraints were violated again. Replication, or near-replication of at least 10% of the experimental budget was used to address surrogate optimism. The chosen formulation E4 was then produced in an independent confirmatory batch.

Table 2. Bayesian formulation-selection variables and experimental search space.

Variable	Symbol	Experimental range	Design rationale
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Dry-film thickness	DFT	70–120 μm	Screening variable; fixed at 90 ± 5 μm in confirmation
Graphene oxide fraction	G	0–0.15 wt%	Barrier tortuosity versus aggregation
IPDI microcapsules	M	0–12 wt%	Crack-triggered sealing capacity
ZIF-8@Ca-type microfillers	Z	0–8 wt%	pH-responsive active-protection reservoir
MCM-41/PEI/PSS nanocontainers	N	0–3 wt%	Controlled inhibitor release
Dynamic-bonding component	H	0–10 mol%	Local repair and stress relaxation
Curing ratio	R	0.90–1.10 of stoichiometric	Crosslink density, mobility, and adhesion

3.1 Durability-quality index and robustness analysis

The durability-quality index (DQI) is a dimensionless decision indicator that combines the electrochemical control, physical integrity, preservation of the product, and economic value. To ensure that each component was treated on [0,1] and had a specific engineering bound, beneficial metrics were scaled up and cost-type metrics were scaled down. The baseline weighting used here was 45% electrochemical control, 45% physical/product integrity and 10% economic value.

$$\text{DQI} = 0.25 Z^* + 0.20 C^* + 0.15 A^* + 0.15 S^* + 0.15 Y^* + 0.10 V^* \quad (1)$$

$$x^*\text{benefit} = \text{clip}[(x - L)/(U - L), 0, 1] \quad (2)$$

$$x^*\text{cost} = \text{clip}[(U - x)/(U - L), 0, 1] \quad (3)$$

In this instance, Z^* is the normalized log10 low frequency impedance, and C^* is the normalized inverse corrosion-rate response, A^* the adhesion retention, S^* the inverse underfilm-corrosion response, Y^* the retention of the net value per tonne of product and V^* the normalized net value per tonne. The robustness was evaluated by the $\pm 20\%$ perturbation of each DQI weight with subsequent re-normalization, and by 10,000 Monte Carlo draws accounting for the uncertainty in the technical and economic-inputs. Rank robustness was a priori defined as ability to maintain the preferred rank in at least 80% of the uncertainty runs. Open circuit stabilization in 3.5 wt% NaCl followed by electrochemical impedance spectroscopy (EIS) with a sinusoidal potential disturbance of 10 mV rms over a frequency range of 100 kHz to 0.01 Hz was conducted. The measurement of the intact, fresh damage, and healed states were performed at certain post-damage times. Data acquisition and validation were based on the principles of ISO 16773-2 [19]. The responses for Bode and Nyquist were reported and low-frequency impedance and equivalent circuit diagnostics were left in place to be interpreted. Polarization resistance/potentiodynamic measurements were made according to ASTM G59, and the corrosion current density was then determined as a linear function of penetration rate with ASTM G102 [20] and [21]. The evaluated steel was used throughout with its equivalent weight $\text{EW} = 27.92 \text{ g eq}^{-1}$ and density $\rho = 7.87 \text{ g cm}^{-3}$.

$$\text{CR} (\text{mm y}^{-1}) = 0.00327 \times i_{\text{corr}} (\mu\text{A cm}^{-2}) \times \text{EW} (\text{g eq}^{-1}) / \rho (\text{g cm}^{-3}) \quad (4)$$

Because impedance spans several orders of magnitude, healing efficiency was evaluated in the logarithmic domain:

$$\eta_Z (\%) = 100 \times [\log_{10}|Z|_{\text{healed}} - \log_{10}|Z|_{\text{scratched}}] / [\log_{10}|Z|_{\text{intact}} - \log_{10}|Z|_{\text{scratched}}] \quad (5)$$



3.2 Accelerated exposure, adhesion, morphology, and bond compatibility

The scribed panels were tested for 720 h under neutral salt fog test per ASTM B117-19 [22]. Scribe corrosion and delamination were tested by ASTM D1654 and ISO 4628-8, respectively, and rusting and blistering were tested by ASTM D610 and ASTM D714 [23]–[25] [28]. To differentiate scribe creep from edge effects, a fixed imaging geometry and scale references was used. The pull-off adhesion performance before and after ageing was evaluated by applying a fixed ASTM D4541-22 adhesion protocol with the controlled fixture size, adhesive, scoring practice, loading rate, cure, substrate and instrument [26]. Representative SEM micrographs and original TGA/DTG traces for the investigated E4 material are provided in Supplementary Data S3–S4 and feature capsule morphology, shell continuity, nanofiller dispersion, cross sections of coatings, damage/healing morphology, and thermal response. Reinforcing bar bond compatibility, using the same E4 formulation as in the corrosion program, was assessed in the comparative framework of ASTM A944 [27] with three independent specimens for each surface condition/size combination tested in No. 6, No. 8 and No. 10 groups. Recorded outputs included failure load (kip), loaded-end slip (in) at failure and residual slip (in) after unloading. Three different mechanically different termination/failure modes were used to interpret the post-test response: steel yielding, bar pullout, and concrete splitting. Specimen level specimen records include the labels bar pullout for 6E-2 and concrete splitting for 6E-4, which were not generally extrapolated to the ones for which they are not explicitly recorded. When steel controlled response occurs, the tensile stress in the bars exceeds yield before substantial bond slip occurs, termed as yielding; when bond slip dominates the force transfer, significant bar slip occurs until the bond fails, which is termed as pullout; when the radial bond stresses cause the formation of diagonal or longitudinal cracks in the concrete and loss of confinement, the response is termed as concrete splitting. There were two factors: surface-condition and bar size, separated by a two factor ANOVA. Under ASTM E4-24 [35] a system of quality assurance for forces was introduced. A full-range verification certificate was generated covering the entire force range of the force applied during the bond program which included the maximum measured failure load = 40.064 kip, and is attached to Supplementary Data S7. Five independent coated coupons were used for the confirmatory comparison for each of the E1–E4 groups. Experimental endpoints are presented as mean mean $\pm$ standard deviation, and the raw data and 95% confidence intervals are provided in Supplementary Data S2. Multiple observations of EIS were not considered to be replicates, but rather within-specimen correlated measurements. The analyses of the specimen-level data were conducted using one-way ANOVA at $\alpha = 0.05$ for the main comparison between E1 and E4 and between E2 and E4. Two factor ANOVA (surface condition $\times$ bar size) was used to analyze the reinforcing-bar data with three independent specimens in each surface-condition/size cell. Failure mechanism, response, and variability were used as criteria for effect interpretation and not only statistical significance. Green-cost analysis has been developed on the basis of a tonne of the product protected. Avoided rework/rejection (AR), avoided claims/storage loss (AC), achievable price premium (PM) and incremental coating cost (IC) were retained as accounting elements. Net value per tonne (NV) and annual net gain (ANG) were calculated as:

$$NV = AR + AC + PM - IC \quad (6)$$

$$ANG = Qp \times NV \quad (7)$$



Avoided material and emissions were calculated independently from financial value to preserve accounting clarity:

$$M_{\text{avoided}} = Q_p \times L_{\text{avoided}} \quad (8)$$

$$E_{\text{avoided}} = M_{\text{avoided}} \times EF_{\text{steel}} \quad (9)$$

For a central industrial scenario, it was assumed that the industrial plant has a capacity of 580,000 t y⁻¹ and that 30% of the output is protected, which means $Q_p = 174,000$ t y⁻¹. Emission factors were estimated for route-sensitive emissions, based on steel production intensities reported by the World Steel Association. Figure 2 depicts the stage-gated process of formulation selection, confirmatory testing, DQI robustness, green-cost analysis and adoption decision.

4. RESULTS

The integrated decision analysis was able to clearly distinguish the five surface states. E4 achieved DQI = 0.91 and a suitability score of 0.96, followed by E3, E2, E1, and B0 (Table 3). It is not a single endpoint type but reflects also at the same time electrochemical suppression, damage recovery, adhesion retention, preservation of the products and the net value.

Table 3. Integrated durability-quality and suitability scores.

Code	DQI	Suitability score	Primary comparative characteristic
B0	0.08	0.00	Unprotected baseline
E1	0.41	0.18	Passive barrier with pronounced scratch sensitivity
E2	0.58	0.46	Barrier reinforcement with modest autonomous recovery
E3	0.73	0.71	Effective crack-triggered sealing
E4	0.91	0.96	Integrated barrier, active protection, sealing, and adhesion retention

The ranking was stable under weight perturbation and under Monte Carlo analysis. E4 was ranked #1 in 94% of the weight-perturbation runs between $\pm 20\%$ and 91% of the Monte Carlo runs of 10,000. In both analysis the E4 DQI uncertainty interval was more than 80% robust (0.88–0.94). The electrochemical response was progressively enhanced with the use of conventional epoxy, to the hybrid E4 architecture (Table 4). E4 reached $\log_{10}|Z|_{0.01\text{Hz}} = 11.59 \pm 0.08$ and $i_{\text{corr}} = 0.065 \pm 0.004 \mu\text{A cm}^{-2}$. Using Eq. (4), the calculated corrosion rate was 7.54×10^{-4} mm y⁻¹. This corresponds to a 97.6% decrease in the corrosion rate calculated on E1 and a low frequency impedance increase of 5.91 decades (impedance ratio of around 8.13×10^5).

Table 4. Electrochemical performance and calculated corrosion rate (n = 5 per coated group; mean $\pm$ SD).

Code	$\log_{10} Z _{0.01\text{Hz}}$	$i_{\text{corr}} (\mu\text{A cm}^{-2})$	CR (mm y ⁻¹)	CR reduction vs E1
B0	3.25 ± 0.15	12.40 ± 0.95	0.14385	—
E1	5.68 ± 0.22	2.70 ± 0.20	0.03132	Reference
E2	7.32 ± 0.18	0.68 ± 0.05	0.00789	74.8%
E3	9.82 ± 0.14	0.21 ± 0.02	0.00244	92.2%
E4	11.59 ± 0.08	0.065 ± 0.004	0.000754	97.6%

The corrosion rate was calculated from i_{corr} using ASTM G102 with $EW = 27.92$ g eq⁻¹ and $\rho = 7.87$ g cm⁻³.



The omnibus formulation effect was very significant for both low-frequency impedance ($F_{3,16} = 1285.47$, $p < 0.001$) and corrosion-current density ($F_{3,16} = 692.98$, $p < 0.001$). The barrier separation and electrochemical separation between E4 and the reference systems were therefore significant. The 720h chloride-exposure results confirmed the electrochemical ranking. The underfilm corrosion for E4 was 0.35 ± 0.04 mm after 24 h, with $95 \pm 2\%$ adhesion and $93 \pm 3\%$ microcrack closure (Table 5). The results for the conventional epoxy E1 were: 7.80 ± 0.45 mm underfilm corrosion, $22 \pm 3\%$ adhesion retention and no measurable crack closure. E2 and E3 were located between the passive barrier reinforcement and autonomous damage repair.

Table 5. Salt-fog, adhesion-retention, and microcrack-closure results (n = 5; mean $\pm$ SD).

Code	Underfilm corrosion after 720 h	Adhesion retention	Microcrack closure after 24 h	Performance interpretation
B0	General rusting	N/A	0%	Unprotected deterioration
E1	7.80 ± 0.45 mm	$22 \pm 3\%$	0%	Passive barrier; severe defect propagation
E2	3.60 ± 0.28 mm	$61 \pm 5\%$	$8 \pm 2\%$	Barrier-dominant response
E3	1.20 ± 0.12 mm	$82 \pm 4\%$	$68 \pm 4\%$	Effective localized healing
E4	0.35 ± 0.04 mm	$95 \pm 2\%$	$93 \pm 3\%$	Integrated protection and recovery

Omnibus ANOVA confirmed strong formulation effects for underfilm corrosion ($F_{3,16} = 750.80$, $p < 0.001$), adhesion retention ($F_{3,16} = 377.04$, $p < 0.001$), and microcrack closure ($F_{3,16} = 1424.54$, $p < 0.001$). The results of electrochemical, visual-corrosion, adhesion, and healing tests suggest that E4 performance was multi-dimensional and not only a result of one test. An aggressive-storage program was performed over a 90-day period that evaluated direct mass loss and maximum pit depth along with engineering derived effective cross-section and load-bearing retention. The lowest mass loss (0.35%) and the deepest pit depth (24 μ m) were observed for E4. The values were 94.0% lower mass loss and 94.1% lower maximum pit depth compared to that of B0 (Table 6).

Table 6. Measured and derived preservation indicators after 90-day aggressive storage.

Indicator	B0	E1	E2	E3	E4	E4 vs B0
Mass loss (%)	5.80	2.10	1.10	0.72	0.35	94.0% lower
Maximum pit depth (μ m)	410	165	95	58	24	94.1% lower
Effective cross-section retained (%)	91.5	95.8	97.2	98.6	99.2	+7.7 percentage points
Load-bearing retention (%)	90.9	95.4	96.8	98.4	99.1	+8.2 percentage points

Mass loss and pit depth are direct exposure measurements; cross-section and load-bearing retention are engineering derivations from the measured deterioration response.

The reinforcing-bar bond data set consisted of 18 individual specimens, comprising three No. 6, three No. 8 and three No. 10 reinforcing bars in each of the two surface conditions (E4 and galvanized). All replicate values, group means and standard deviations are reported in Table 7. Mean failure loads for E4-coated bars were 28.571 ± 2.482 kip (No. 6), 28.827 ± 1.602 kip (No. 8), and 32.237 ± 1.367 kip (No. 10); corresponding galvanized-bar means were 28.467 ± 2.704 ,



29.518 $\pm$ 1.021, and 36.329 $\pm$ 3.415 kip. The two-way ANOVA showed that the bar size (2-way) effect on failure load was significant, $F(2,12) = 11.71$, $p = 0.0015$, but the surface condition (E4-versus-galvanized) main effect was not significant ($F(1,12) = 2.15$, $p = 0.169$), nor was the surface condition $\times$ size interaction ($p = 0.270$). The bar-size effect was significant for loaded-end slip, $F(2,12) = 43.58$, $p < 0.001$, and there was no main effect of surface-condition ($p = 0.355$). There were no significant main effects for either surface condition ($p = 0.577$) or bar size ($p = 0.444$), nor for the interaction between surface condition and bar size ($p = 0.397$) on residual slip after unloading. Specimen level record shows that 6E-2 failed through bar pullout at 25.710 kip and 0.0319 in loaded-end slip while 6E-4 failed through concrete splitting at 30.148 kip and 0.0353 in loaded-end slip. This contrasts the fact that equal load-slip magnitudes can end via various mechanisms so should be read in the context of the failure mode observed. Failure loads of all measured failures were within the range specified by the full-range ASTM E4 verification certificate recorded in Supplementary Data S7, with a maximum value of 40.064 kip. The bond results are interpreted relative to each other based upon ASTM A944 and are not translated into design bond strengths.

Table 7. Replicate ASTM A944-framework bond-test dataset for E4-coated and galvanized reinforcing bars, including documented specimen-level failure modes.

Bar condition	Specimen	Failure load (kip)	Slip at load (in)	Slip after unloading (in)	Recorded failure mode
E4-coated	6E-2	25.710	0.0319	0.0044	Pullout
E4-coated	6E-4	30.148	0.0353	0.0051	Concrete splitting
E4-coated	6E-5	29.855	0.0446	0.0031	—
E4-coated	No. 6 mean $\pm$ SD	28.571 $\pm$ 2.482	0.0372 $\pm$ 0.0066	0.0042 $\pm$ 0.0010	—
E4-coated	8E-1	30.468	0.0185	0.0033	—
E4-coated	8E-2	28.745	0.0200	0.0039	—
E4-coated	8E-3	27.268	0.0179	0.0040	—
E4-coated	No. 8 mean $\pm$ SD	28.827 $\pm$ 1.602	0.0188 $\pm$ 0.0011	0.0038 $\pm$ 0.0004	—
E4-coated	10E-1	32.911	0.0147	0.0040	—
E4-coated	10E-2	30.663	0.0119	0.0039	—
E4-coated	10E-3	33.136	0.0148	0.0029	—
E4-coated	No. 10 mean $\pm$ SD	32.237 $\pm$ 1.367	0.0138 $\pm$ 0.0016	0.0036 $\pm$ 0.0006	—
Galvanized	G-1	31.584	0.0335	0.0041	—
Galvanized	6G-3	27.059	0.0332	0.0046	—
Galvanized	6G-4	26.757	0.0273	0.0046	—
Galvanized	No. 6 mean $\pm$ SD	28.467 $\pm$ 2.704	0.0313 $\pm$ 0.0035	0.0044 $\pm$ 0.0003	—
Galvanized	8G-1	28.565	0.0170	0.0017	—
Galvanized	8G-2	30.595	0.0124	0.0015	—
Galvanized	8G-3	29.394	0.0201	0.0030	—
Galvanized	No. 8 mean $\pm$ SD	29.518 $\pm$ 1.021	0.0165 $\pm$ 0.0039	0.0020 $\pm$ 0.0008	—
Galvanized	10G-3	35.559	0.0140	0.0025	—
Galvanized	10G-4	33.365	0.0153	0.0029	—
Galvanized	10G-5	40.064	0.0211	0.0194	—



Bar condition	Specimen	Failure load (kip)	Slip at load (in)	Slip after unloading (in)	Recorded failure mode
Galvanized	No. 10 mean $\pm$ SD	36.329 $\pm$ 3.415	0.0168 $\pm$ 0.0038	0.0083 $\pm$ 0.0096	—

Note: Specimen-specific failure modes were recorded for 6E-2 (pullout) and 6E-4 (concrete splitting). No failure mode is assigned to other specimens unless supported by an explicit specimen record.



Table 8. Mechanical classification of bond-test termination and failure modes used in the present interpretation.

Mode	Mechanical description	Principal controlling factors
Steel yielding	Tensile stress in the reinforcing bar reaches the steel yield level, producing permanent elongation before dominant slip develops along the bonded length.	Favored when anchorage/bond capacity is high relative to steel yield resistance; influenced by bar grade, bonded length, and confinement.
Bar pullout	The steel-concrete interface loses its ability to transfer increasing force and the bar slips from the concrete with limited macro-splitting of the surrounding block.	Reduced mechanical interlock, interfacial defects or voids, insufficient bonded length, surface condition, and inadequate confinement can promote pullout.
Concrete splitting	Radial bearing stresses generated by the deformed bar produce longitudinal or diagonal cracking in the surrounding concrete, followed by loss of confinement and abrupt bond resistance.	Governed principally by concrete cover, transverse confinement, concrete tensile resistance, bar diameter/rib geometry, and the magnitude of radial bond stress.

Failure-mode interpretation was carried out in conjunction with load-slip response and not just from peak load. The difference is illustrated directly in the documented specimens with E4 coating, 6E-2 with pullout at 25.710 kip and 0.0319 in loaded-end slip, and 6E-4 with concrete splitting at 30.148 kip and 0.0353 in loaded-end slip. Pullout is an interface-controlled termination whereas concrete splitting is a stress-controlled termination, as it is determined through radial bond stresses and loss of concrete confinement, and steel yielding is another steel-controlled termination. Therefore, it cannot be assumed that similar peak loads indicate the same bond mechanism, and coating compatibility is best argued based on load, slip and failure mode observed. Representative SEM micrograph and original TGA/DTG traces of the studied E4 material are shown along with scale bars, instrument settings and curve records in Supplementary Data S3–S4. These provide primary supporting characterization of the E4 architecture and provide documentation of capsule morphology, nanofiller distribution, coating cross sectional features, damage/healing morphology and thermal response. A documented MCI CorShield dataset was maintained as an external benchmark, the mean value of each of three trials (AET Test Report #05-01412) reported as an ASTM A944-99 standard. The benchmark coating was close to the control at the two lower applied loads, and had lower elongation at the loaded end at 10,400 lb, 15,000 lb and 20,900 lb, with a 32.9% lower elongation at the highest applied load. This external dataset is presented in Table 9 and has no contribution to the E4 DQI or ANOVA nor is it used in the corrosion-performance calculations.

Table 9. External ASTM A944-99 bond benchmark for MCI CorShield versus control (mean of three trials; AET Test Report #05-01412) [34].

Applied load, lb (kg)	Control loaded-end elongation, in (mm)	MCI CorShield loaded-end elongation, in (mm)	Relative change vs control
2,800 (1,270)	0.053 (1.35)	0.050 (1.27)	5.7% lower
7,300 (3,311)	0.090 (2.29)	0.091 (2.31)	1.1% higher
10,400 (4,717)	0.220 (5.59)	0.134 (3.40)	39.1% lower
15,000 (6,804)	0.293 (7.44)	0.171 (4.34)	41.6% lower
20,900 (9,480)	0.319 (8.10)	0.214 (5.44)	32.9% lower

Note: This is a data-set not used in the E4 experiment. The loaded-end elongation values were manually entered from the provided data sheet and checked with Cortec/AET reference and are not used for the E4 DQI or statistical comparisons. The laboratory durability response was then



expressed in a plant-scale decision-making scenario based on the operational inputs defined. The protected volume was 174,000 t y⁻¹ with a nominal plant capacity of 580,000 t y⁻¹ and a selective treatment of 30%. The gross benefit was USD 63 t⁻¹, due to avoided rework / rejection, avoided claims / storage loss and high durability price premium. The central net value was USD 32 t⁻¹, projected annual net gain was USD 5.568 million (Table 10) and the incremental coating cost was USD 31 t⁻¹.

Table 10. Central green-cost calculation.

Variable	Central value	Calculation basis
Nominal plant capacity	580,000 t y ⁻¹	Industrial scenario basis
Protected high-durability share	30%	Selective conversion of product lines
Protected product volume	174,000 t y ⁻¹	$580,000 \times 0.30$
Incremental coating cost	USD 31 t ⁻¹	Materials + application + curing + inspection + QC
Avoided rework and rejection	USD 29 t ⁻¹	Reduced cleaning, reclassification, cutting loss, rejected dispatch
Avoided claims and storage loss	USD 11 t ⁻¹	Reduced complaints and storage downgrading
Price premium	USD 23 t ⁻¹	High-durability product value
Net value added	USD 32 t ⁻¹	$29 + 11 + 23 - 31$
Annual net gain	USD 5.568 million y ⁻¹	$174,000 \times 32$
Avoided steel replacement/rework	2,088 t y ⁻¹	$174,000 \times 1.2\%$
Reference-factor avoided emissions	3,132 tCO ₂ e y ⁻¹	$2,088 \times 1.5 \text{ tCO}_2\text{e t}^{-1}$

Positive economic value was maintained in the three adoption cases throughout the sensitivity analysis (Table 11). The central case generated USD 5.57 million y⁻¹, and the high-adoption case generated USD 10.67 million y⁻¹. The steel-production range of 0.70–2.32 tCO₂ t⁻¹ was used as a steel-production route to separate route-sensitive carbon estimates (Table 12) due to the material difference between the steel-production routes of scrap-EAF and BF-BOF production.

Table 11. Economic sensitivity for selective plant adoption.

Scenario	Protected volume (t y ⁻¹)	Incremental cost (USD t ⁻¹)	Gross benefit (USD t ⁻¹)	Net value (USD t ⁻¹)	Annual net gain (USD million y ⁻¹)
Conservative	87,000	35	48	13	1.13
Central	174,000	31	63	32	5.57
High adoption	232,000	29	75	46	10.67

Table 12. Avoided-material and route-sensitive emission sensitivity.

Scenario	Avoided material loss	Avoided steel (t y ⁻¹)	Reference estimate at 1.5 tCO ₂ e/t	Route-sensitive CO ₂ range (0.70–2.32 tCO ₂ /t)
Conservative	0.8%	696	1,044 tCO ₂ e y ⁻¹	487–1,615 tCO ₂ y ⁻¹
Central	1.2%	2,088	3,132 tCO ₂ e y ⁻¹	1,462–4,844 tCO ₂ y ⁻¹



High adoption	1.6%	3,712	5,568 tCO2e y-1	2,598–8,612 tCO2 y-1
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5. DISCUSSION The comparative ranking is in accordance with the functional hierarchy designed for the coating systems. E1 used passive barrier action, and exhibited high propagation following artificial damage. E2 decreased transport because of the tortuosity induced by the graphene-oxide, but did not exhibit a significant autonomous response at the defect. E3 increased the sealing of cracks by the introduction of crack triggered sealing, and as a result greatly improved the behavior of the scribes and the recovery of microcracks. E4 integrated all of these mechanisms along with responsive inhibitor delivery and dynamic-network recovery, resulting in the highest impedance, corrosion current smallest, adhesion retention highest, and underfilm-corrosion distance smallest. The interpretation is in line with the general research of microcapsule-based repairs, nanocontainer release, and pH-responsive ZIF systems. Specifically, the electrochemical resistance of the E4 is retained and/or restored in the event of local coating damage, and the controlled loading of microcapsules can ensure a practical level of adhesion. The present data take it one step further by bringing together the barrier, sealing, inhibitor and dynamic-bond mechanisms in a single, bayesian selected formulation. The E4 result is strong because of the consensus in independent performance areas. The 5.91 decade advantage over E1 occurred when the corrosion rate has been reduced by 97.6%, underfilm corrosion was reduced from 7.80 to 0.35 mm, adhesion retention was improved from 22% to 95%, and the microcrack closure was increased from 0% to 93%. Although these endpoints examined various aspects of coating behaviour, such as electrolyte transport, electrochemical kinetics, lateral corrosion propagation, interface strength, and physical recovery, the same formulation hierarchy was arrived at. The storage indicators based on 90 days also support the same conclusion at product-preservation level. E4 decreased the overall material loss and localized pitting without compromising the loss of derived cross section. This is industrially important as handling / storage corrosion can impact downstream cleaning / reclassification, claims and replacement demand as well as appearance. This replica reinforcing bar test was added to the study to provide a direct application level compatibility layer due to the fact that the same E4 formulation was tested in the corrosion program on the epoxy-coated specimens. The mean of E4-coated failure load was +0.4% higher than the mean of the corresponding galvanized loads for No. 6 and No. 8 groups, while it was -11.3% lower for No. 10 group. Dominant variance in failure load was due to bar size ($p = 0.0015$) and the main effect of surface-condition was not significant ($p = 0.169$); the surface-condition $\times$ size interaction was not significant. The loading at the ends (loaded-end slip) also depended mainly on the bar size. Mechanical interpretation was used to differentiate steel yielding from the two primary bond-controlled mechanisms, namely, pullout and concrete splitting. Yielding means that anchorage is sufficient enough to mobilize steel plasticity before significant loss of dominant bond; pullout represents progressive loss of force transfer at the bond interface; and splitting represents that the bond stresses are radial and exceed the tensile/confinement resistance of the surrounding concrete. The failure loads tested were within the force range specified by the full range ASTM E4 verification certificate (Supplementary Data S7). MCI CorShield benchmark is an external comparator only. As a group, the bond data provide an indication of compatibility of E4 coated reinforcement for the bar sizes tested, but do not imply that ASTM A944 response is a structural design bond strength. The DQI explicitly overcomes this issue and gives the user a method for aggregating the disparate performance measures without hiding away the effect of the component tests. Robustness was assessed directly, rather than it being assumed, as decision weights may have an impact on a composite index. E4 consistently dominated in the weight-perturbation runs (94%), and in Monte Carlo



draws, DQI was held back at 0.88-0.94. There was a fair amount of variation in the weighting and structure of the inputs with only slight changes in the ranking overall. This robustness is important to plant decision making, since engineering, commercial and environmental goals rarely do stand still. A formulation that is more preferred in all aspects (economics, physical integrity and electrochemical performance) is more defensible in the process of scaling up than a formulation that is chosen based on one metric. The green-cost model translates technical durability into an easily explainable operating decision. It is deliberately additive and auditable: the coating spend is compared against avoided rework/rejection, avoided claims/storage loss and price premium, and the avoided material and carbon are quantified in parallel. The central scenario shows an USD 32 t⁻¹ net value with an USD 5.568 million annual gain, which clearly indicates that corrosion protection is not only a cost of an additional coating, but a value-preserving investment. The environmental outcome is also very dependent on the avoided material and steel-production route. Separating the material-loss fraction from the route-specific emissions factor allows the model to be modified to account for different BF-BOF, DRI-EAF or scrap-EAF supply conditions without needing to modify the experimental corrosion data. This establishes a direct link between the performance of the coating, green-cost management, and emissions accounting. Five independent coated replicates of each E1–E4 formulation are incorporated in the validation chain, along with electrochemical testing and accelerated-exposure testing, direct storage-preservation measurements, primary SEM/TGA characterization and an independent 18-specimen reinforcing bar bond program for the same E4 formulation. The bond force measurements are within range of the full range ASTM E4 verification certificate archive, and specimen records contain a record of load, slip, variation and observed failure mode. This evidence structure, in combination with the 10,000-draw uncertainty analysis and explicit green-cost sensitivity cases, is a traceable basis for pilot-line transfer and further field/plant verification.



6. CONCLUSIONS

In this study, an integrated experimental and decision framework for selection and validation of self-healing nanoencapsulated epoxy coatings for reinforcement of steel was established. Bayesian-guided screening efficiently addressed the interaction of the formulation variables and confirmatory testing under fixed DFT and standardized conditions of exposure differentiated the performance of the conventional, barrier-enhanced, microcapsule-healing, and hybrid systems. The formulation E4 was the most preferred formulation in the designed space. It achieved $\log_{10}|Z|_{0.01\text{Hz}} = 11.59 \pm 0.08$, $i_{\text{corr}} = 0.065 \pm 0.004 \mu\text{A cm}^{-2}$, and a calculated corrosion rate of $7.54 \times 10^{-4} \text{ mm y}^{-1}$. The corrosion-rate reduction was 97.6% when compared with E1. E4 was only able to prevent underfilm corrosion to $0.35 \pm 0.04 \text{ mm}$ after 720 h salt fog, and maintained $95 \pm 2\%$ adhesion and $93 \pm 3\%$ microcrack closure. The mass loss during the 90-day preservation test was found to be 0.35% with maximum pit depth of 24 μm . The integrated DQI of 0.91 was not affected by the sensitivity and uncertainty analysis, and under weight-perturbation, E4 stayed in first place in 94% of the runs, and in 91% of the Monte Carlo draws. The central green-cost scenario produced USD 32 t⁻¹ net value and USD 5.568 million y⁻¹ of projected gains for 174,000 t y⁻¹ of protected product; route-sensitive avoided emission calculations correlated the durability gain with avoided material loss and achieved steel-production carbon intensity. As a group, the results indicate that the E4 hybrid architecture (diffusion barrier reinforcement, crack triggered sealing, pH responsive inhibitor delivery, and dynamic network recovery) performed best when it came to corrosion-control and maintained its first rank decision position as well as with weight and input uncertainty. Application level testing of E4-coated reinforcing bars for No. 6, No. 8 and No. 10 groups revealed that bar size ($p = 0.0015$) is a significant factor in determining failure-load response while the E4 versus galvanized surface-condition main effect is not significant ($p = 0.169$). Slip and failure mode also played an important role in the interpretation of the peak load and were also observed at the specimen level, identifying two mechanisms: pullout and concrete splitting. The corrosion, bond, robustness, and green-cost outcomes are therefore a single auditable evidence chain for pilot scale implementation and industrial adoption planning of the E4 system; the outcomes of ASTM A944 remain comparative, not as a structural design value.



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