EJSIT
European Journal of Science, Innovation and Technology
ISSN: 2786-4936
www.ejsit-journal.com
Volume 5 | Number 5 | 2025

Experimental Design and Optimization of a Novel Hybrid Epoxy-Based Coating for Corrosion Resistance Using Response Surface Methodology

Oghenekowho, P. A.*, Oreko, B. U., Ajuwa, C. I.
Mechanical Engineering Department,
Federal University of Petroleum Resources, Effurun, Nigeria

ABSTRACT

The problem of corrosion of carbon steel in acidic environment remains a serious problem in industrial usage, causing a shortening of service life, and higher maintenance expenses. In this paper, a novel hybrid manganese oxide (MnO_2) and coconut husk nanoparticles (CHNP) have been developed and optimized to enhance corrosion resistance. The experimental runs were designed and optimized using Response Surface Methodology (RSM) where MnO_2 and CHNP loadings were the key variables. The weight loss method in varied concentration of a simulated acidic media (0.5 – 2.5 M H_2SO_4 solution) was used to evaluate the corrosion behaviour. Findings showed the hybrid coatings to have excellent protective efficiency of over 94% and a drastic reduction in corrosion rate occurred (35.42 mm/yr in uncoated steel to 1.8424 mm/yr in optimum hybrid coating). The sufficiency of the RSM models was statistically confirmed and the coefficients of determination were high ($R^2 = 0.99$). It is shown that agro-waste-derived CHNP, mixed with MnO_2, is a cost-effective, environmentally friendly, and high-performance reinforcer of epoxy coating when used in a harsh acidic condition.

Keywords: Hybrid epoxy coating, Carbon steel, Corrosion resistance, Coconut husk nanoparticles, Manganese oxide, Response Surface Methodology

INTRODUCTION

Metallic corrosion, particularly in carbon steel, is a significant concern in the construction, petrochemical, and manufacturing industries. Carbon steel easily corrodes when it is subjected to acidic conditions, causing structural failures, environmental and economic losses (Kania, 2023; Zehra, Mobin, & & Aslam, 2022; Joseph, SBanjo, Afolalu, & Babaremu, 2022; Aryai, Baji, & Mahmoodian, 2022; Menga, et al., 2023). Corrosion protective coatings are considered one of the best solutions to this problem (Kania, 2023; Chopra, Ola, Dhayal, & Shekhawat, 2022; Sesia, Spriano, Sangermano, & Ferraris, 2023), and the most commonly used protective coatings are the epoxy-based systems (Anwar & Li, 2024). This is due to their high adhesion, resistance to chemicals, and low cost (Sreehari, Sethulekshmi, & & Saritha, 2022; Yuan, et al., 2022; Rahman & Akhtarul Islam, 2022). However, traditional epoxy paints tend to be brittle and cannot perform well in harsh environments over time (Chen, et al., 2024; Fu, Wu, Li, Xu, & Wang, 2022; Chen, et al., 2022; Bratasyuk, Latyshev, & Zuev, 2023).

In order to overcome these shortcomings, hybrid nanocomposite coatings have been explored. One way to enhance the barrier performance, toughness, and corrosion resistance of an epoxy matrix is to incorporate nanoparticles into the matrix (Zhang, et al., 2023; Samardžija, Alar, Špada, & Stojanović, 2022; Iyer, Nayak, Hiremath, Heckadka, & Jaideep, 2023; Esmailzadeh, Tammari, Safarpour, Razavian, & Pezzato, 2024; Srinivasa Perumal, Selvarajan, Mathan Kumar, & Shriguppikar, 2025). Among the inorganic additives, metallic oxide nanoparticles exhibit superior redox activity and surface stability, which can be utilised to enhance the passive barrier properties of coatings (Wang, et al., 2022; Ezzeddin & Al-khalidi,

* Corresponding Author

2024; Gnanavelbabu, Vinothkumar, & Prahadeeswaran,, 2024; Okon, et al., 2025). Meanwhile, bio-based reinforcements, such as agro-wastes, are sustainable alternatives, cost-effective and enhance mechanical reinforcement, promoting the development of eco-friendly materials (Oisakede & Sadjere, 2022; Joshi, Bajpai, & Mukhopadhyay, 2023; Opadoyin, Fayomi, & Atiba, 2024).

Design of coating formulations has key experimental optimization techniques that are essential in the selection of the formulations to maximise protective capabilities. Response Surface Methodology has become a potent statistical instrument to optimize the parameters of experiments and reduce the number of tests, but with a high level of predictive models (Elkelawy, et al., 2024; Dubey, Prasad, Kumar Singh, & Nayyar, 2022; Kocakulak, et al., 2023). Although there has been growing interest in nanocomposite coatings, only a few researchers have reported on the synergistic application of agro-waste-derived nanoparticles and inorganic reinforcements with epoxy coatings, especially with RSM to optimize their use. In this work, experimental design, preparation and optimization of hybrid epoxy coating reinforced with a combination of MnO_2 and CHNP are therefore emphasized. The developed coating was then immersed in an acidic medium (H_2SO_4), and weight loss technique was used to determine the corrosion resistance. The goals include identifying the best ratio of the novel hybrid coating, testing its protective effectiveness against the corrosion of carbon steel and testing the predictive potential of the RSM model.

MATERIALS AND METHODS

Materials

Carbon steel substrates (composition: C = 0.67%, Mn = 0.58%, Si = 0.06%, P = 0.01%, S = 0.01%, Cu = 0.21%, Fe = balance) with dimensions 4 cm × 4 cm × 5 cm were used. Epoxy resin and curing agent, distilled water, and acetone were procured from Onitsha Chemical market, Nigeria. MnO_2 nanoparticles (purity of 99.4%) were obtained from Kermel Chemical International, while coconut husk was sourced locally, dried, ground, and processed into nanoparticles.

Preparation of Nanoparticles

Coconut husk was washed to remove any debris, sun-dried for three days, pulverized, and subjected to mechanical ball milling for 78 hours. The resulting powder was sieved using a magnetic sieve of 75 µm mesh. MnO_2 nanoparticles were used as received, with particle sizes averaging 65-100 nm.

Hybrid Coating Formulation

Different ratios of MnO_2 and CHNP as obtained from the RSM experimental design were dispersed into epoxy resin using mechanical stirring to ensure homogeneity. The hardener was added in a ratio 2:1 according to the manufacturer's recommendation, and the mixture was applied onto polished carbon steel specimens using the dip technique. Coated samples were left to cure at room temperature for about 48 hours.

Experimental Design Using RSM

A Central Composite Design (CCD) was employed using MINITAB 17 software. The independent variables were MnO_2 in wt. %, CHNP in wt. % and H_2SO_4 in mol/dm^{3,} while the response was corrosion rate. A total of 20 experimental runs were generated, and the results were analysed using ANOVA to determine model significance.

Weight Loss Method of Corrosion Testing

At room temperature, samples were immersed for a 30-day period in the different H_2SO_4 concentration to assess the loss in mass and hence calculate the extent of corrosion. Initial and final weights of the specimens were taken and corrosion rate (C_R) was calculated using equation 1.

$$C_R = \frac{K\ X\ W}{A\ X\ D\ X\ T} \tag{1}$$

Where C_R = corrosion in mm/yr
K = 87600
W = weight loss in grams (g)
A = total area of exposed surface in cm^2
D = density of material in g/cm^3 (D for steel is $7.85 g/cm^3$)
T = time of exposure in hours

The optimum blend to achieve minimum corrosion rate for the coating will then be analysed by carrying out optimization using the MINITAM software. The optimal result obtained was then validated experimentally and used to calculate the protective efficiency of the developed coating using equation 2.

$$PE\ (\%) = \frac{C_{R,uncouted} - C_{R,couted}}{C_{R,uncouted}}\ X\ 100 \tag{2}$$

RESULTS AND DISCUSSION

Weight Loss Data

The weight loss calculation and corrosion rate calculated using equation 1 is shown in Table 1. It can be seen that the third experimental run with 4.18 wt % CHNP and 1.81 Wt % MnO_2 in 1 mol/dm^3 H_2SO_4 experienced the lowest corrosion rate while the sixth experimental run with 1.18 wt % CHNP and 4.81 Wt % MnO_2 in 2 mol/dm^3 H_2SO_4 experienced the highest corrosion rate. This suggests that the effect of CHNP and MnO_2 in the coating is inversely proportional.

Table 1: Weight Loss and Calculated Corrosion Rate

RUN	H_2SO_4 (mol/dm^3)	CHNP (wt %)	MnO_2 (wt %)	W1 (g)	W2 (g)	W1-W2 (g)	CORR RATE (mm/yr)
1	1	1.81	1.81	61.779	57.964	3.815	3.6955
2	2	1.81	1.81	65.167	60.874	4.293	4.1586
3	1	4.18	1.81	71.765	69.47	2.295	2.2231
4	2	4.18	1.81	65.649	62.813	2.836	2.7472
5	1	1.81	4.18	67.506	61.574	5.932	5.7462
6	2	1.81	4.18	58.824	52.48	6.344	6.1453
7	1	4.18	4.18	68.403	64.689	3.714	3.5977
8	2	4.18	4.18	58.824	54.827	3.997	3.8718
9	0.5	3	3	66.216	62.35	3.866	3.7449
10	2.5	3	3	60.593	56.32	4.273	4.1392
11	1.5	1	3	64.264	58.389	5.875	5.6910
12	1.5	5	3	61.131	58.688	2.443	2.3665
13	1.5	3	1	63.329	60.87	2.459	2.3820
14	1.5	3	5	70.174	64.453	5.721	5.5418
15	1.5	3	3	62.421	58.339	4.082	3.9542
16	1.5	3	3	64.125	60.252	3.873	3.7517
17	1.5	3	3	66.778	63.054	3.724	3.6074
18	1.5	3	3	57.343	53.352	3.991	3.8660
19	1.5	3	3	61.745	57.956	3.789	3.6703
20	1.5	3	3	62.156	58.26	3.896	3.7740

RSM Optimization Results

The results of ANOVA, as shown in Table 2, supported the quadratic models as significant ($p < 0.05$). The model summary table shown in Table 3 gives the R^2 value of the model as 99.13%. This implies that the model was able to explain over 99% of the data supplied. It can also be seen in Table 2 that the 2-way interaction for the two reinforcements used in developing the hybrid coating had significant effect on the corrosion rate.

Table 2: ANOVA Table

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	23.3846	2.5983	126.50	0.000
Linear	3	22.8384	7.6128	370.64	0.000
H_2SO_4	1	0.3743	0.3743	18.22	0.002
CHNP	1	12.1813	12.1813	593.06	0.000
MnO_2	1	10.2824	10.2824	500.61	0.000
Square	3	0.2336	0.0779	3.79	0.047
H_2SO_4* H_2SO_4	1	0.0603	0.0603	2.94	0.117
CHNP*CHNP	1	0.1472	0.1472	7.17	0.023
MnO_2*MnO_2	1	0.0877	0.0877	4.27	0.066
2-Way Interaction	3	0.3085	0.1028	5.01	0.023
H_2SO_4*CHNP	1	0.0005	0.0005	0.03	0.874
H_2SO_4* MnO_2	1	0.0125	0.0125	0.61	0.454
CHNP* MnO_2	1	0.2955	0.2955	14.39	0.004
Error	10	0.2054	0.0205		
Lack-of-Fit	5	0.1255	0.0251	1.57	0.316
Pure Error	5	0.0799	0.0160		
Total	19	23.5900			

Table 3: Model Summary

S	R-sq	sq(adj)	R-sq(pred)
0.143317	99.13%	98.35%	95.22%

The regression equation of uncoded units developed by the software is given in equation 3.

$$\begin{aligned} \text{CORR RATE} = {} & 3.499 - 0.032\, H_2SO_4 - 0.797\, \text{CHNP} + 0.907\, MnO_2 + 0.193\, H_2SO_4{*}H_2SO_4 + \\ & + 0.0722\, \text{CHNP}{*}\text{CHNP} + 0.0557\, MnO_2{*}MnO_2 - 0.0139\, H_2SO_4{*}\text{CHNP} - \\ & - 0.0666\, H_2SO_4{*}MnO_2 - 0.1369\, \text{CHNP}{*}MnO_2 \end{aligned} \tag{3}$$

The contour plots of corrosion rate are given in Figure 1. It can be observed that corrosion rate reduced as the quantity of CHNP increased but increased with increased MnO_2 concentration. It can also be seen that although corrosion rate increased as the acid concentration increased, the increment was gentler as MnO_2 quantity increased in the coating combination. It can be deducted that the CHNP in the coating had more positive effect on corrosion rate but the presence of MnO_2 acted as a stabilizer in acid medium. The surface plot given in Figure 2 showed that increment in corrosion rate as the acid concentration increased was mild, indicating that the coating performed well in acid medium.

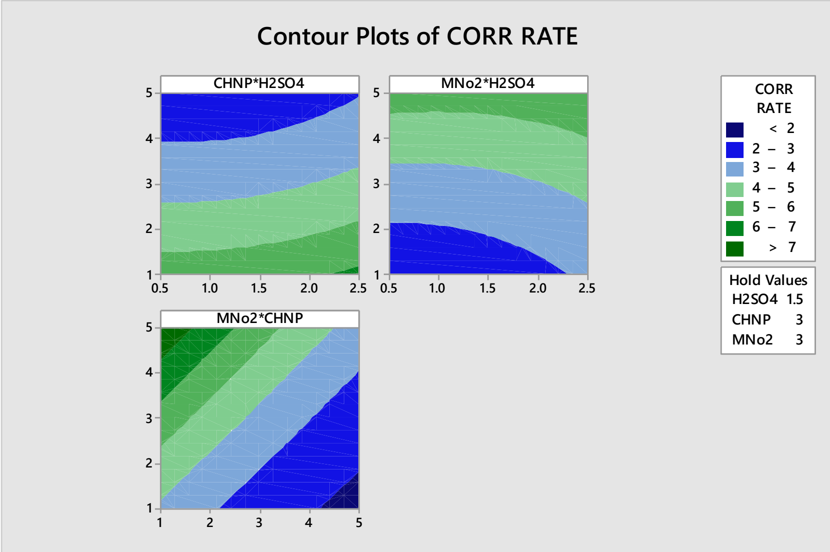

Figure 1: Contour Plot for Corrosion Rate

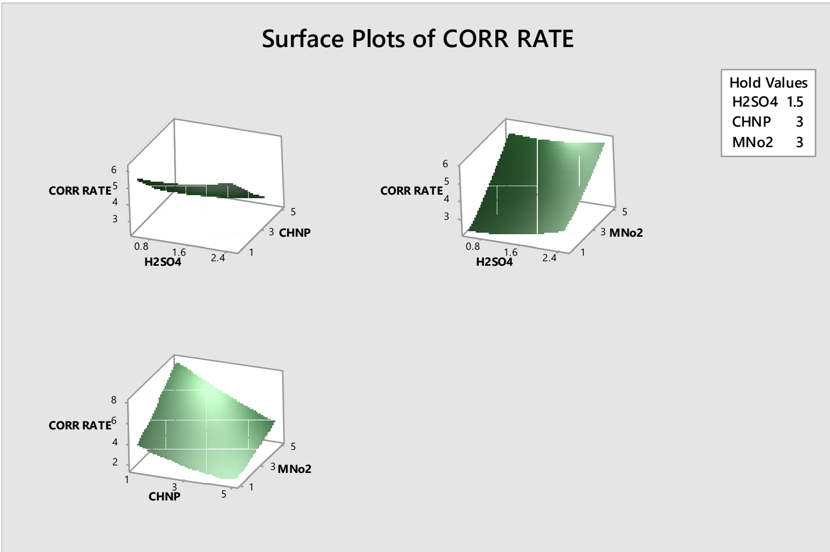

Figure 2: Surface Plots for Corrosion Rate

It can be seen in the optimisation plot given in Figure 3 that at a desirability of 100%, an optimal blend of 4.65 wt %CHNP and 1,5 wt % MnO_2 immersed in 0.7 M H_2SO_4 would experience a corrosion rate of just 1.8401 mm/yr. These values were validated experimentally and the result obtained for corrosion rate was 1.8424 mm/yr which means there was just a 0.12% difference between the experimental result and that predicted by the software.

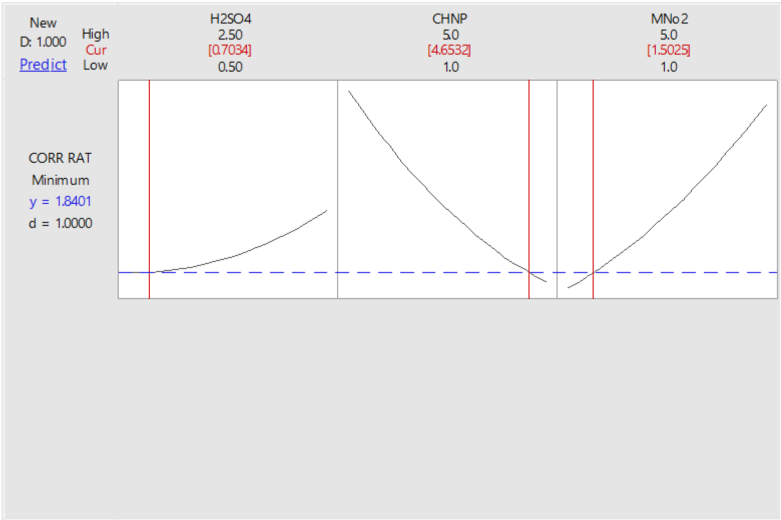

Figure 3: Optimisation Plot

Protective Efficiency

The calculated corrosion rates of the blank/uncoated and optimal specimen is given in Table 4.

Table 4: Protective Efficiency of Optimal Sample

SAMPLE	W1 (g)	W2 (g)	W1-W2 (g)	CORR RATE (mm/yr)	P.E (%)
Blank	59.246	22.678	36.568	35.4228	0
Optimal	58.64	56.738	1.902	1.8424	94.8

The calculated protective efficiency of the optimal combination was found to be 94.8%. This shows the synergy of combining MnO_2 and CHNP in the inhibition of corrosive assault on carbon streel.

Summary of Findings

The developed novel hybrid coating showed excellent performance. This could be due to enhanced particle packing, barrier stability and high adhesion of nanoparticles in the epoxy matrix. The efficiency of almost 95% obtained is quite impressive. It is worthy of note that agro-waste (CHNP) offers a sustainable, low-cost cost and environmentally friendly route to the development of advanced coatings

CONCLUSIONS

An innovative hybrid MnO_2 and CHNP reinforced epoxy coating has been developed and optimized with the help of RSM. The 0.7 M $H2SO_4$ weight loss tests established a high corrosion resistance of the hybrid coating, as compared to uncoated carbon steel. The optimal formulation was found to show protective efficiency of approximately 95% and a reduction in the corrosion rate from 35.42 mm/yr (uncoated) to 1.84 mm/yr (hybrid). RSM models were statistically significant and effective in predicting coating performance.

REFERENCES

Anwar, S., & Li, X. (2024). A review of high-quality epoxy resins for corrosion-resistant applications. *J Coat Technol Res, 21*, 461-480. doi:10.1007/s11998-023-00865-5

Aryai, V., Baji, H., & Mahmoodian, M. (2022). Failure assessment of corrosion affected pipeline networks with limited failure data availability. *Process Safety and Environmental Protection, 157*, 306-319. doi:10.1016/j.psep.2021.11.024

Bratasyuk, N. A., Latyshev, A. V., & Zuev, V. V. (2023). Water in Epoxy Coatings: Basic Principles of Interaction with Polymer Matrix and the Influence on Coating Life Cycle. *coatings, 14*(1). doi:10.3390/coatings14010054

Chen, Q., Wang, C., Yu, S., Song, Z., Fu, H., & An, T. (2022). Low-temperature mechanical properties of polyurethane-modified waterborne epoxy resin for pavement coating. *International Journal of Pavement Engineering, 24*(2). doi:10.1080/10298436.2022.2099853

Chen, Z., Liu, X., Chen, H., Li, J., Wang, X., & Zhu, J. (2024). Application of epoxy resin in cultural relics protection. *Chinese Chemical Letters, 35*(4). doi:10.1016/j.cclet.2023.109194

Chopra, I., Ola, S. K., Dhayal, V., & Shekhawat, D. S. (2022). Recent advances in epoxy coatings for corrosion protection of steel: Experimental and modelling approach-A review. *Materials Today: Proceedings, 62*(3), 1658-1663. doi:10.1016/j.matpr.2022.04.659

Dubey, A., Prasad, R. S., Kumar Singh, J., & Nayyar, A. (2022). Optimization of diesel engine performance and emissions with biodiesel-diesel blends and EGR using response surface methodology (RSM). *Cleaner Engineering and Technology, 8*, 1-11. doi:10.1016/j.clet.2022.100509

Elkelawy, M., El Shenawy, E. A., Bastawissi, H. A., Shams, M. M., Balasubramanian, D., Anand, V., & Alwetaishi, M. (2024). Predictive modeling and optimization of a waste cooking oil biodiesel/diesel powered CI engine: An RSM approach with central composite design. *Scientific Reports, 14*(1), 1-13. doi:10.1038/s41598-024-77234-8

Esmailzadeh, M., Tammari, E., Safarpour, T., Razavian, S. M., & Pezzato, L. (2024). Anti-corrosion effect of chitin and chitosan nanoparticles in epoxy coatings. *Materials Chemistry and Physics, 317*. doi:10.1016/j.matchemphys.2024.129097

Ezzeddin, B., & Al-khalidi, M. (2024). An investigation into the effect of using different metal oxide nanoparticles on the anti-corrosion properties of coatings: a comparative study. *Moroccan Journal of Chemistry, 12*(2), 657-675. doi:10.48317/IMIST.PRSM/morjchem-v12i2.43008

Fu, Q., Wu, S., Li, C., Xu, J., & Wang, D. (2022). Delamination and chip breaking mechanism of orthogonal cutting CFRP/Ti6Al4V composite. *Journal of Manufacturing Processes, 73*, 183-196. doi:10.1016/j.jmapro.2021.11.015

Gnanavelbabu, A., Vinothkumar, E., & P. M. (2024). Investigation on corrosion behaviour of metal oxide nanoparticles reinforced magnesium composites by salt spray and immersion corrosion test methods. *Advances in Materials and Processing Technologies*, 1-25. doi:10.1080/2374068X.2024.2402971

Iyer, T., Nayak, S. Y., Hiremath, A., Heckadka, S. S., & Jaideep, J. P. (2023). Influence of TiO2 nanoparticle modification on the mechanical properties of basalt-reinforced epoxy composites. *Cogent Engineering, 10*(1). doi:10.1080/23311916.2023.2227397

Joseph, O. O., SBanjo, S., Afolalu, S. A., & Babaremu, K. O. (2022). Global and economic effects of corrosion - An overview. *AIP Conf. Proc, 2437*. doi:10.1063/5.0092286

Joshi, R., Bajpai, P. K., & Mukhopadhyay, S. (2023). Processing and performance evaluation of agro wastes reinforced bio-based epoxy hybrid composites. *Proceedings of the*

Institution of Mechanical Engineers, Part L: Journal of Materials: Design and Applications, 237(2). doi:10.1177/14644207221118686

Kania, H. (2023). Corrosion and Anticorrosion of Alloys/Metals: The Important Global Issue. *Coatings, 13*(2). doi:10.3390/coatings13020216

Kocakulak, T., Halis, S., Ardebili, S. M., Babagiray, M., Haşimoğlu, C., Rabeti, M., & Calam, A. (2023). Predictive modelling and optimization of performance and emissions of an auto-ignited heavy naphtha/n-heptane fueled HCCI engine using RSM. *Fuel, 333*. doi:10.1016/j.fuel.2022.126519

Menga, A., Kanstad, T., Cantero, D., Bathen, L., Hornbostel, K., & Klausen, A. (2023). Corrosion-induced damages and failures of posttensioned bridges: A literature review. *Structural Concrete, 24*(1), 84-99. doi:10.1002/suco.202200297

Oisakede, M. O., & Sadjere, E. G. (2022). Analysis of the Wear Resistance of Epoxy-Agro Waste Nanoparticle Coating. *NIPES Journal of Science and Technology Research, 4*(2), 289 - 305.

Okon, K., Ekeke, I. C., Maduabuchi, C. A., Ayogu, I. I., Azeez, T. O., & Akalezi, C. O. (2025). Recent advances in the use of metal oxides as corrosion inhibitors: a review. *KOM – Corrosion and Material Protection Journal, 69*, 14-33. doi:10.2478/kom-2025-0003

Opadoyin, P. O., Fayomi, O. S., & Atiba, J. O. (2024). Agro-Waste Nanofiller as a Reinforcer for Service Life Improvement of Coated Sample. *International Conference on Science, Engineering and Business for Driving Sustainable Development Goals (SEB4SDG).* Omu-Aran, Nigeria: IEEE.

Rahman, M., & Akhtarul Islam, M. (2022). Application of epoxy resins in building materials: progress and prospects. *Polym. Bull., 79*, 1949–1975. doi:10.1007/s00289-021-03577-1

Samardžija, M., Alar, V., Špada, V., & Stojanović, I. (2022). Corrosion Behaviour of an Epoxy Resin Reinforced with Aluminium Nanoparticles. *Coatings, 12*(10). doi:10.3390/coatings12101500

Sesia, R., Spriano, S., Sangermano, M., & Ferraris, S. (2023). Natural Polyphenols and the Corrosion Protection of Steel: Recent Advances and Future Perspectives for Green and Promising Strategies. *metals, 13*(6), 1070-1097. doi:10.3390/met13061070

Sreehari, H., Sethulekshmi, A. S., & & Saritha, A. (2022). Bio Epoxy Coatings: An Emergent Green Anticorrosive Coating for the Future. *Macromolecular Materials and Engineering, 307*(8).

Srinivasa Perumal, K., Selvarajan, L., Mathan Kumar, P., & Shriguppikar, S. (2025). Enhancing mechanical and morphological properties of glass fiber reinforced epoxy polymer composites through rutile nanoparticle incorporation. *Prog Addit Manuf, 10*, 831-848. doi:10.1007/s40964-024-00675-0

Wang, J., Wu, S., Ma, L., Zhao, B., Xu, H., Ding, X., & Zhang, D. (2022). Corrosion resistant coating with passive protection and self-healing property based on Fe3O4-MBT nanoparticles. *Corrosion Communications, 7*, 1-11. doi:10.1016/j.corcom.2021.12.005

Yuan, S., Zhao, X., Jin, Z., Liu, N., Zhang, B., Wang, L., . . . Hou, B. (2022). Fabrication of an environment-friendly epoxy coating with flexible superhydrophobicity and anti-corrosion performance. *Colloids and Surfaces A: Physicochemical and Engineering Aspects, 633*(1). doi:10.1016/j.colsurfa.2021.127545

Zehra, S., Mobin, M., & & Aslam, J. (2022). 1 - An overview of the corrosion chemistry. *Environmentally Sustainable Corrosion Inhibitors*, 3-23.

Zhang, D., Huang, Y., Xu, L., Tao, C., Yang, X., & Wang, X. (2023). Synergistic effects of nanoparticle geometric shape and post curing on carbon-based nanoparticle reinforced epoxy coatings: Characterization, microstructure, and adhesion properties. *Progress in Organic Coatings, 185*. doi:10.1016/j.porgcoat.2023.107929