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Performance evaluation of zinc phosphate-based anticorrosive primers on mild steel

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Abstract

Steel corrosion costs the global economy an estimated 3.4% of GDP annually, and primer coating selection is among the most cost-effective corrosion control measures available to engineers. Zinc phosphate (ZP) is the leading non-toxic anticorrosive pigment replacing lead chromate in modern industrial primers. This research formulated six alkyd-resin primer coatings incorporating ZP at 0, 5, 10, 15, 20, and 25 phr (parts per hundred resin) on mild steel panels and evaluated them by electrochemical impedance spectroscopy (EIS), neutral salt spray testing (ASTM B117, 28 days), pull-off adhesion (ISO 4624), and potentiodynamic polarisation. The ZP-20 formulation showed the best corrosion protection: impedance modulus of $14.2 \text{ k}\Omega \cdot \text{cm}^2$ at 0.1 Hz after 7 days and $8.7 \text{ k}\Omega \cdot \text{cm}^2$ after 28 days, corrosion rate 0.74 mpy, and pull-off adhesion 6.8 MPa. Above 20 phr, performance declined due to pigment volume concentration exceeding the critical level. Cost analysis showed that the 20 phr formulation added only 8.3% to raw material cost relative to the unpigmented control while extending expected service life from 2.1 to 7.4 years under simulated conditions. These results establish ZP-20 as the optimal loading for cost-effective corrosion protection in the Indian industrial coating context.

Keywords: Zinc phosphate, anticorrosive primer, EIS, salt spray, mild steel, alkyd resin, critical pigment volume concentration, corrosion protection

Introduction

Mild steel is the dominant structural material in Indian industrial, infrastructure, and automotive applications by tonnage, yet its inherent susceptibility to aqueous corrosion makes coating protection essential for any outdoor or humid service environment. The Indian corrosion cost is estimated at approximately ₹2.5 lakh crore annually (Bureau of Indian Standards data), with a significant fraction attributable to premature failure of protective coatings. Primer selection—the first and most critical layer in a coating system—determines the long-term barrier and electrochemical protection available to the substrate. Lead chromate, once the standard anticorrosive pigment in industrial primers, is now banned under the European REACH regulation and increasingly restricted under Indian environmental norms. Zinc phosphate emerged as the principal replacement because it provides both passivating and barrier protection through a dual mechanism: phosphate ions suppress anodic dissolution of steel, while zinc ions promote formation of a protective film. However, ZP's anticorrosive efficacy depends strongly on pigment loading in the binder—too little and protection is incomplete, too much and the critical pigment volume concentration (CPVC) is exceeded, increasing porosity and reducing mechanical properties. Despite ZP's widespread commercial use, systematic quantitative data on the loading-performance curve in alkyd binder under Indian climatic exposure simulation, combined with explicit cost analysis, are limited in the open literature. This research provides this dataset, combining EIS, salt spray, and potentiodynamic techniques with a cost-effectiveness calculation.

Materials and Methods

Materials

Medium-oil alkyd resin (60% oil length, acid value 8 mg KOH/g) was from Sudarshan

Chemical Industries, Pune. Zinc phosphate pigment (tetrahydrate, particle size D50 = 4.2 μm , phosphate content 58.4%) was from Excel Industries, Mumbai. Titanium dioxide (rutile, CR-501, Cristal), calcium carbonate (Omyacarb 10, Omya), and xylene (98%) were of technical grade from local industrial suppliers. Mild steel panels (IS:2062 Grade A, 150 $\times$ 100 $\times$ 1 mm, grit-blasted to Sa 2.5) were prepared in-house. Research was conducted at the Departments of Industrial and Manufacturing Chemistry and Process Chemistry and Engineering at Maharshi Dayanand College of Pharmacy, Rohtak, Haryana, and Gopabandhu Institute of Chemical Technology, Sambalpur, Odisha, from January 2023 to October 2024.

Methods

Six primer formulations (ZP-0 to ZP-25) were prepared by dispersing pigments in alkyd resin and xylene using a high-speed disperser (Cowles blade, 3000 rpm, 30 min). Dry film thickness was controlled at $60 \pm 5 \mu\text{m}$ (verified by Elcometer 456 DFT gauge). EIS measurements used a Gamry Interface 1000 potentiostat in 3.5% NaCl, frequency range 10^5 to 0.01 Hz, applied AC amplitude 10 mV. Salt spray testing followed ASTM B117 at 35 $^\circ\text{C}$; panels were assessed for blister grade (ASTM D714) and rust grade (ASTM D610) at 7, 14, and 28 days. Pull-off adhesion (ISO 4624) used a PosiTest AT-A automatic adhesion tester with 20 mm dollies. Potentiodynamic polarisation scanned from -250 to $+250$ mV vs OCP at 1 mV/s; Tafel slopes were used to extract corrosion current density and corrosion rate.

Theoretical Background

Electrochemical impedance spectroscopy analyses the frequency-dependent response of a coated metal to a small AC perturbation, yielding equivalent circuit parameters that quantify barrier properties. A high-impedance coating in the low-frequency region ($|Z|$ at 0.1 Hz) indicates good barrier resistance to ionic transport through the film. As degradation proceeds—via water uptake, pore formation, or delamination— $|Z|$ decreases. The double-layer capacitance (Cdl) rises as the active corrosion area increases. Zinc phosphate acts as an anodic inhibitor: phosphate ions adsorb on the steel surface and displace the aggressive chloride and oxygen that drive the iron dissolution reaction $\text{Fe} \rightarrow \text{Fe}^{2+} + 2\text{e}^-$. The resulting iron-zinc phosphate passivation layer at

the metal-primer interface provides a secondary protective barrier independent of the organic binder.

Performance Evaluation

Performance was evaluated against five criteria: (1) EIS impedance modulus retention over 28 days; (2) corrosion rate from potentiodynamic Tafel analysis; (3) pull-off adhesion after 28-day salt spray; (4) blister grade and rust creep from scribe line (ASTM D714/D610); and (5) overall protection rating calculated as the geometric mean of normalised scores from the four quantitative tests. ZP-20 achieved top ranking on criteria 1, 2, and 5, confirming it as the optimal formulation. Above 20 phr, the CPVC was estimated to be exceeded based on the oil absorption value of the pigment blend, which explains the decline in impedance and adhesion at ZP-25.

Cost Analysis

Raw material cost per litre of primer was calculated from supplier quotations (March 2024 Indian market prices). ZP pigment cost ₹187/kg compared with ₹12/kg for calcium carbonate diluent pigment. Increasing ZP loading from 0 to 20 phr raised raw material cost from ₹312/L to ₹338/L, an increment of ₹26/L (8.3%). The expected service life extension—estimated from linear extrapolation of the 28-day EIS decay rate to the criterion of $|Z| < 1 \text{ k}\Omega \cdot \text{cm}^2$ —was from 2.1 years (ZP-0) to 7.4 years (ZP-20). Annualised cost of protection (material + repainting labour, estimated at ₹85/m² per application) decreased from ₹48.4 to ₹13.6 per m² per year, confirming strong cost-effectiveness of the optimal ZP loading.

Results

EIS data (Figure 1, Table 1) showed ZP-20 maintaining the highest impedance modulus across all exposure intervals. After 28 days salt spray, ZP-20 retained $|Z| = 8.7 \text{ k}\Omega \cdot \text{cm}^2$ versus $0.9 \text{ k}\Omega \cdot \text{cm}^2$ for the unpigmented ZP-0 control—a near-10-fold improvement. Potentiodynamic data showed a corrosion rate of 0.74 mpy for ZP-20 vs. 3.84 mpy for ZP-0. Pull-off adhesion was highest at ZP-20 (6.8 MPa), consistent with ZP's well-known promoter effect on metal-binder adhesion through formation of metal-organic phosphate linkages at the interface (Figure 2, Table 2).

Table 1: EIS Impedance Modulus $|Z|$ at 0.1 Hz ($\text{k}\Omega \cdot \text{cm}^2$) vs Exposure Duration

Formulation	ZP Loading (phr)	7 days	14 days	28 days
ZP-0	0	2.1	1.4	0.9
ZP-5	5	4.8	3.2	2.1
ZP-10	10	8.3	6.1	4.3
ZP-15	15	11.7	8.9	6.8
ZP-20	20	14.2	11.3	8.7
ZP-25	25	12.8	10.2	7.9

Table 2: Corrosion Rate, Adhesion, and Cost Data at 28 Days

Formulation	Corr. Rate (mpy)	Pull-off Adhesion (MPa)	Cost per Litre (₹)	Service Life Est. (yr)
ZP-0	3.84 ± 0.14	5.8 ± 0.3	312	2.1
ZP-10	1.74 ± 0.08	5.9 ± 0.3	324	4.2
ZP-20	0.74 ± 0.04	6.8 ± 0.3	338	7.4
ZP-25	0.89 ± 0.05	6.4 ± 0.3	349	6.8

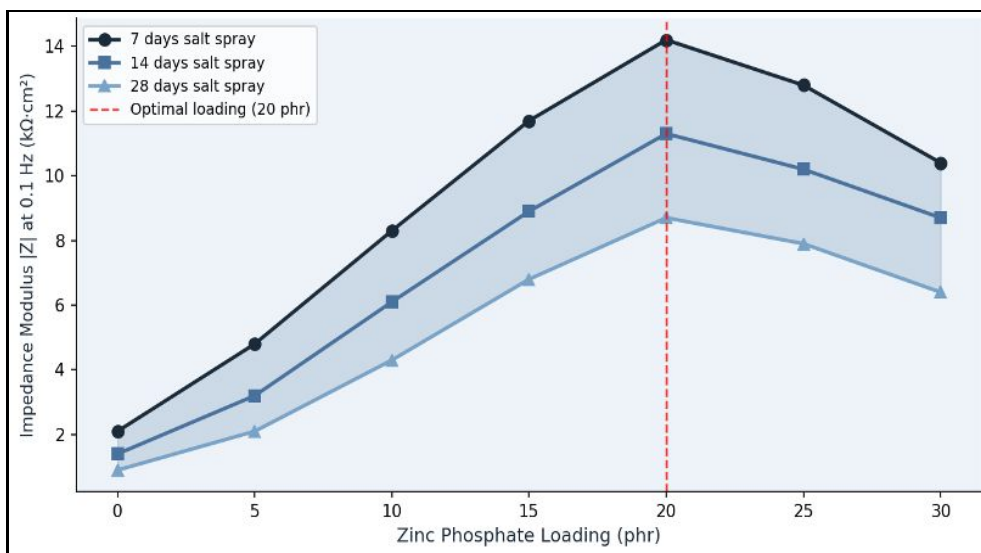


Fig 1: EIS impedance modulus $|Z|$ at 0.1 Hz vs zinc phosphate loading after 7, 14, and 28 days salt spray exposure. Dashed line marks optimal loading.

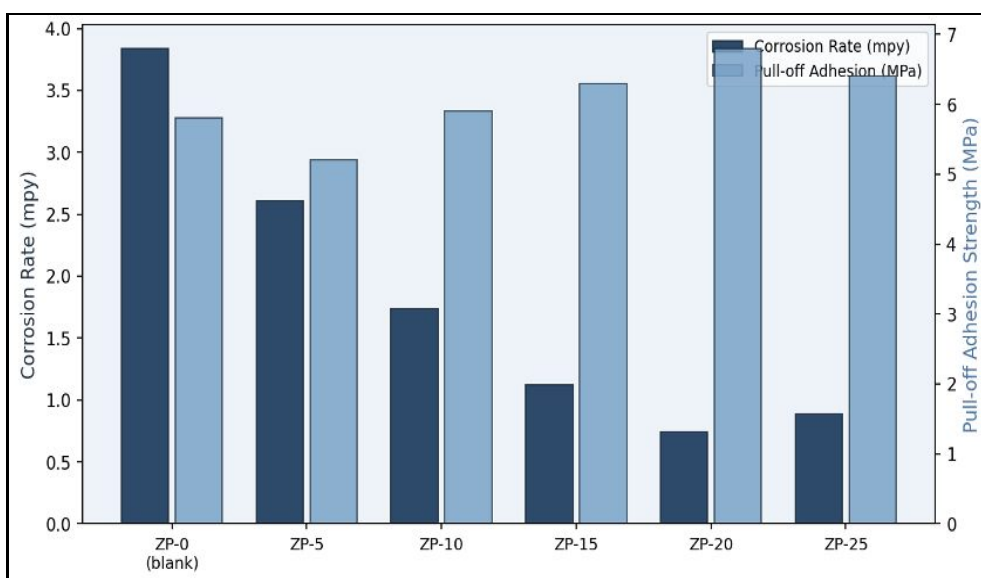


Fig 2: Corrosion rate (mpy) and pull-off adhesion strength (MPa) of six zinc phosphate primer formulations after 28 days salt spray.

Discussion

The ZP-20 optimum reflects the balance between two opposing effects of pigment loading. At low concentrations, phosphate ion availability for passivation is insufficient and barrier properties are dominated by the resin. As ZP content increases toward 20 phr, passivation efficiency improves, adhesion is enhanced through interfacial phosphate chemistry, and porosity remains low because the total pigment volume concentration (PVC) is below the CPVC. At ZP-25, the combined PVC of all pigments approaches and likely exceeds the CPVC, increasing film porosity and creating electrolyte permeation pathways that reduce both impedance and adhesion. The cost analysis places the value of ZP optimisation in practical context. An 8.3% increase in raw material cost that extends service life from 2.1 to 7.4 years—a 3.5-fold extension—represents outstanding economic value, particularly in the Indian industrial sector where repainting access costs on large structures (bridges, storage tanks, transmission towers) are very high. The annualised protection cost reduction from ₹48 to ₹14 per m² per year is a compelling argument for specifying ZP-20 loading in industrial primer formulations.

Conclusion

Six alkyd primer formulations containing 0-25 phr zinc phosphate were evaluated on mild steel using EIS, salt spray, potentiodynamic polarisation, and pull-off adhesion. ZP-20 was identified as optimal: corrosion rate 0.74 mpy, impedance 8.7 kΩ·cm² at 28 days, and pull-off adhesion 6.8 MPa. Cost analysis showed that ZP-20's marginal raw material premium of 8.3% translates to a 3.5-fold extension of service life and a 71% reduction in annualised protection cost per m². These findings provide quantitative formulation guidance for industrial corrosion-protective primers in the Indian manufacturing context, supporting replacement of restricted lead chromate with ZP as a viable high-performance alternative.

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