Impact of Contact Angle on Corrosion Behaviour of Polydimethylsiloxane and Nanofillers Based Nanocomposite Coated Galvanized Iron

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Abstract: This study focuses on formulating polymer nanocomposite solutions based on polydimethylsiloxane (PDMS) and nanofillers (ZnO and SiO_2) for coating galvanized iron (GI) via the sol-gel dip coating method, with a particular emphasis on investigating their corrosion resistance. Various weight percentages (0, 2, 4, 6, 8, and 10) of nanofillers are incorporated into PDMS and xylene solutions to create the nanocomposite coating solution. The resulting solutions are applied onto GI substrates and subjected to characterization using scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDS) and Fourier-transform infrared spectroscopy (FTIR), followed by assessments of wettability and electrochemical responses. SEM with EDS and FTIR analysis reveals surface morphology and chemical composition of the coated surfaces, identifying Si-O-Si groups at 1088 cm^{-1}. Wettability studies ascertain the hydrophobicity of the coatings. Electrochemical analysis demonstrates a reduction in corrosion rate with increasing contact angle. Remarkably, the coating containing 10% nanofillers exhibits the highest contact angle and lowest corrosion rate, suggesting potential applications in the construction, marine, and naval sectors.

Keywords: Sol-gel coating; Polydimethylsiloxane; Nanofillers; Hydrophobicity; Corrosion behavior.

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1. Introduction

Corrosion represents a significant functional challenge in metal components, leading to substantial material and energy losses globally. Research indicates that approximately 1/5th of the energy and an average of 4.2% of gross products are lost each year due to metal corrosion. Current methods to control corrosion, such as inspection, repairing corroded surfaces, applying

protective coatings, and disposing of corroded waste, involve considerable costs. However, the adoption of non-corrosive materials often proves economically unfeasible for basic applications. Therefore, selecting appropriate coating materials to prevent metal corrosion becomes paramount [1][2].

Among the available anti-corrosion methods, organic coatings stand out due to their ease of processability and applicability [3]. Certain organic coatings have the capability to transform metallic surfaces into hydrophobic ones, effectively mitigating corrosion. Hydrophobicity, primarily determined by the contact angle exceeding 90°, can be further enhanced by coating low surface energy materials or increasing surface roughness [4].

Various surface coating methods exist, including electrophoretic, powder, and sol-gel coatings. The sol-gel method is particularly noteworthy for its cost-effectiveness, durability, and minimal environmental impact. This method operates at near-room temperature using liquid precursors, making it suitable for wide-ranging applications such as roofing, piping, valves, and couplings [5]. Despite the widespread use of galvanized iron (GI) sheets in various industries, research on nanocomposite coatings for GI sheets remains limited. This research gap necessitates the exploration of cost-effective and easily applicable coating solutions for GI sheets. Our study aims to address this gap by developing and characterizing PDMS/ZnO/SiO_2-based nanocomposite solutions for GI coating using SEM with EDS, and FTIR. Then, their hydrophobicity and corrosion behavior were assessed through wettability and electrochemical analyses.

2. Experimental details

2.1 Materials

PDMS resin, procured from Dow Chemical, USA, is used as an adhesive. ZnO and SiO_2, procured from Sigma-Aldrich Chemicals Private Limited, Bangalore, in powder forms with an average size of 100-200 nm, are used as nanofillers in the current work. Xylene and GI sheets supplied by Gupta Enterprises, Bangalore, are used as solvents and substrates, respectively.

2.2 Solution synthesis, Substrate preparation and development of coating

The coating process initiates with the blending of PDMS (10 wt. %) and xylene to form solution A, ensuring a uniform mixture through stirring at 50 °C. Solutions B to F are then created by combining xylene with nanofillers (ZnO-X % and SiO2-Y %) in equal proportions, followed by ultrasonication to disperse the nanofillers evenly. Before coating, substrates (3×5 cm^2) undergo cleaning. Cleaning involves immersing the substrate in a 2.5% NaOH solution at 70 ºC for 10 minutes, followed by a water break test to ensure cleanliness. Substrates are then rinsed in demineralized water and dried with a blower to remove any residual moisture. Figure 1(a) illustrates the prepared bare (GI) substrate through stereo-microscopic imaging. Coating involves

dip coating in solutions A-F, followed by drying in an oven at 180 °C for 10 minutes. The resulting uniform coatings (designated as A to F) are suitable for further analysis and application, as depicted in scanning electron microscopy images with a coating thickness of 21.8 μm (Figure 1(b)).

2.3 Electrochemical corrosion test

The corrosion resistivity of the coating was investigated using a potentiostat. The test involved three electrodes: a working electrode (representing the sample), a saturated calomel electrode (used as the reference electrode), and a platinum electrode (acting as the counter electrode). The applied potential ranged from -250 mV to +250 mV. To induce corrosion in a 1 cm2 area of the samples, a 3.5% NaCl solution was utilized. The scanning speed remained constant at 5 mV/s throughout the experiment.

The corrosion rate (CR) in millimetres per year was determined using the formula:

$$CR \text{ (mm/y)} = 3.2\,X10^{-3}\,\frac{Icorr\ X\ A}{\rho} \quad \ldots\ldots (1)$$

Where Icorr represents the corrosion current density in A/cm^2, CR is the corrosion rate in millimetres per year, A is the molar mass, and ρ is the density of galvanised iron.

3. Results and discussion

3.1 Surface morphology and chemical composition

SEM is used for surface morphological analysis to observe the distribution of the nanofillers and the various defects in the coated surfaces. The SEM images of the coated surface-F are shown in Figure 1. The nanofillers' presence and the uniform distribution are observed in the coated surface-F, as shown in Figure 1(c). This will further enhance the corrosion-resistant properties of the coated surface. The agglomeration (Figure 1(d)) of the nanofillers develops roughness. The presence of air pockets in the coated surface-F is also observed in Figure 1(e). These air pockets help minimize the contact area of the solution with the coated surface [6]. A similar observation is noted in Ref. [7]. Further, EDS is also performed to trace the different elements of the nanofillers present in the coated surface. The EDS of the coated surface-F is shown in Figure 1(f). The presence of the Si, Zn and O elements is clearly observed in the coated surface F. The Si element exhibited a higher peak value, which shows more Si element content than the Zn element in the coated surface-F. It may be attributed to the presence of more Si content in the captured region in the EDS analysis due to the accumulation of Si particles at the localized places in the coated surface, as observed Figure 1(d).

FTIR analysis is utilized to determine the chemical composition of coated surfaces by identifying chemical bonds through the absorption of infrared energy. This analysis generates a plot correlating transmittance and wavenumber, as illustrated in Figure 2(a). Peaks on this plot signify distinct functional groups.

Notably, the peaks for all coated samples closely align. Specifically, the peak at 769 cm^{-1} indicates Si-H bonds, while Si-O-Si groups appear at 1088 cm-1. Symmetric stretching of the -CH3- group is evident at 3021.7 cm^{-1} and 2832.4 cm^{-1}. Si-C stretching in Si-CH_3 produces a peak at 843 cm^{-1}, and stretching of the epoxy ring results in peaks at 1605 cm^{-1} and 1340.5 cm^{-1}.

FTIR spectra of all coated samples exhibit similar characteristic PDMS peaks, indicating that the polymer backbone remains chemically intact after nanofiller addition. No new peaks or significant shifts were observed, suggesting that nanofillers are physically incorporated and well-dispersed within the PDMS matrix rather than forming new chemical bonds. The improved corrosion resistance is therefore attributed to morphological modification and barrier enhancement rather than chemical bonding.

3.2 Wettability studies

To improve surface corrosion resistance, it's essential for the coated surface to be hydrophobic, preventing prolonged water contact. Hydrophobicity, indicated by the surface's ability to repel liquid solutions, is evaluated through wettability analysis, specifically by measuring the contact angle between the substrate and liquid droplet. In this study, the wettability of the coated surfaces was evaluated through contact angle measurements, as shown in Figure 2 (b–h) and Table1. For each coated sample, contact angle measurements were performed three times at different locations on the surface to ensure measurement reproducibility and reliability. Results revealed that while the bare GI substrate was initially hydrophilic (contact angle: 83.8°± 2.1°), all coated surfaces exhibited hydrophobic behaviour (contact angle > 90°). Moreover, increasing nanofiller percentage correlated with higher contact angles. For instance, the coated substrate-F displayed the maximum contact angle (139°± 2.3°), 29.9% higher than coated surface-A. This increase limits water contact, enhancing corrosion resistance. The observed trend can be attributed to PDMS's low surface energy and the introduction of nanofillers, which create micro-roughness textures with air pockets (Figure 1(e)), facilitating water droplet rebounding and minimizing contact area.

3.3 Corrosion behaviour

The corrosion rate is an important parameter that signifies how fast or slow a surface's corrosion occurs. The Potentiodynamic polarization tests are conducted for all the coated surfaces and the electrochemical parameters are listed in Table 2. The Tafel plot of each sample is obtained and presented in Figure 3. The corrosion rate of the samples is calculated by Tafel extrapolation method using Gamry software. The calculated values of the corrosion rates for all the coated samples are given in Table 2. It is also observed that the coated surface's corrosion rate decreases with an increase in nanofiller %. The decrease in the corrosion rate is observed from 7.86-80.47 % for the coated surface-B to F compared to the coated surface-A. This may be attributed to the decrease in the

current density (Icorr) of the coated samples. The incorporation of the nanofillers fills the pores of the PDMS, which leads to the nonporous structure, leading to more compact. The compacted nonporous structures operate as a solid barrier against NaCl solution penetration, significantly reducing corrosion. A clear reduction in corrosion rate was observed with increasing nanofiller content. For coating-F, the corrosion rate was measured as (93.2 ± 4.1) × 10□³ mpy, which is significantly lower than that of coating-A. The relatively small standard deviation values indicate good experimental reproducibility. Although coating-D showed a comparatively higher corrosion potential, its corrosion rate did not reach the minimum value due to partial passivation and insufficient nanofiller content to form a fully compact barrier layer. In contrast, higher nanofiller loading in coating-F resulted in superior barrier integrity and corrosion resistance. [8]. The corrosion rate is less for all the coated samples than the bare (GI), which is approximately 68.21 ± 3.8 mpy, showing the influence of the nanofillers on the corrosion resistance [9].

4. Conclusion

PDMS-based nanocomposite solutions are utilized for coating GI sheets, involving nanofiller dispersion. Coated surfaces undergo SEM with EDS, FTIR analysis, alongside wettability and corrosion testing. These evaluations unveil surface morphology and chemical composition. Nanofiller addition elevates contact angles, with coating-F achieving a remarkable 139°. Corrosion rates notably decrease across all surfaces, with surface-F demonstrating superior resistance, attributed to its enhanced contact angle. This highlights its potential for varied engineering applications necessitating corrosion protection, marking a significant advancement in surface coating technology.

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Table 1. Contact angle of Coated Surfaces.

Sample	Contact angle (°)
Bare GI	83.8 ± 2.1
A	107. ± 2.6
B	113.5° ± 2.4°
C	121.2° ± 2.2°
D	128.6° ± 2.0°
E	134.1° ± 2.1°
F	139.0° ± 2.3°

Table 2.EIS parameter for the coated surfaces in 3.5% NaCl solution.

Sample	E_{corr} (V)	I_{corr} ($\times 10^{-6}$ A/cm^2)	Corrosion rate ($\times 10^{-}$3 mpy)
A	-0.207	9.037	477.3 ± 21.5
B	-1.070	8.327	439.8 ± 19.6
C	-1.050	7.486	395.4 ± 17.8
D	-0.114	4.886	262.2 ± 12.4
E	-0.981	2.607	137.7 ± 6.9
F	-0.994	1.765	93.2 ± 4.1

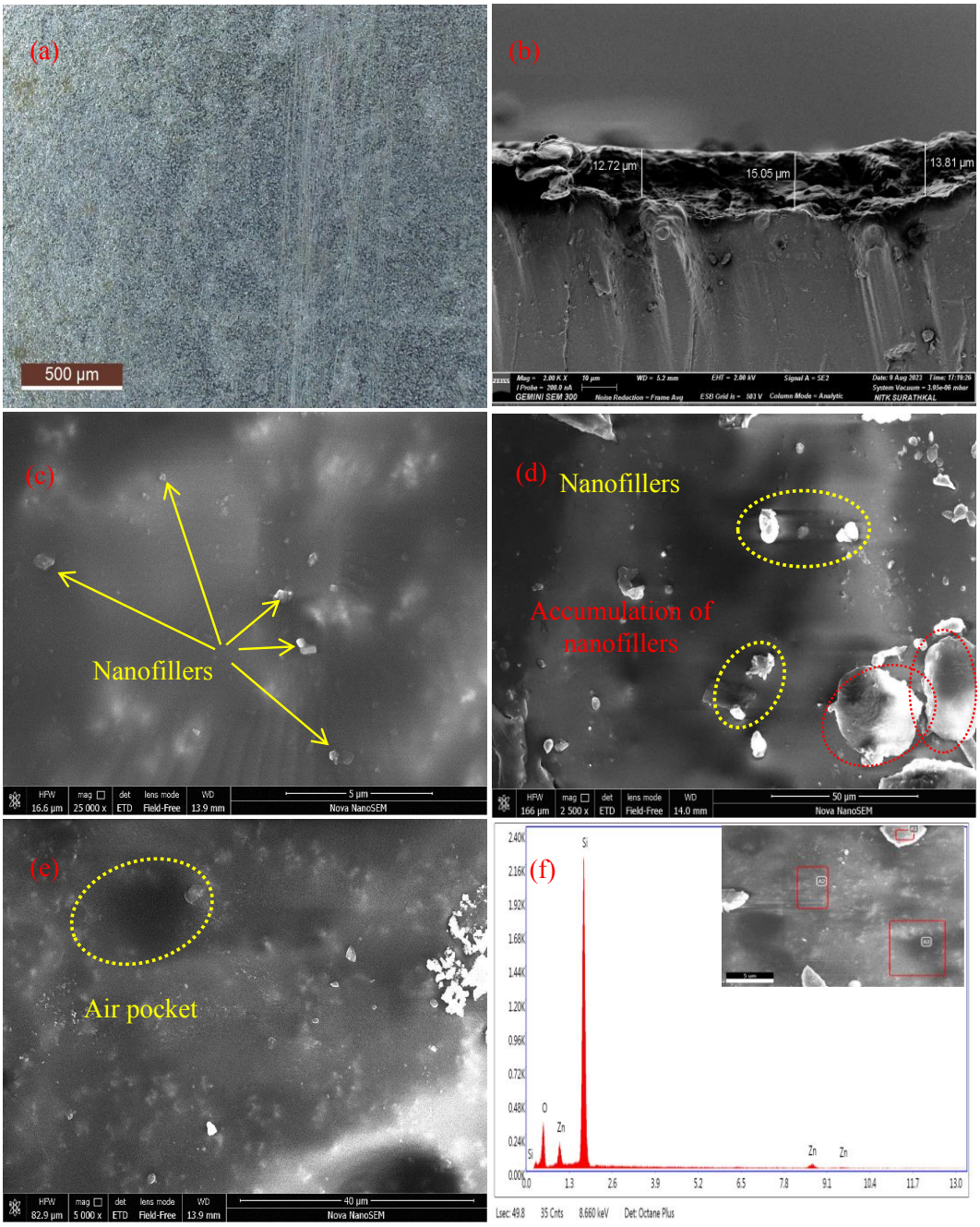

Figure 1. Micrographs(a) Bare GI, (b)Cross section, and(c)-(f) Surface morphology of Coating-F.

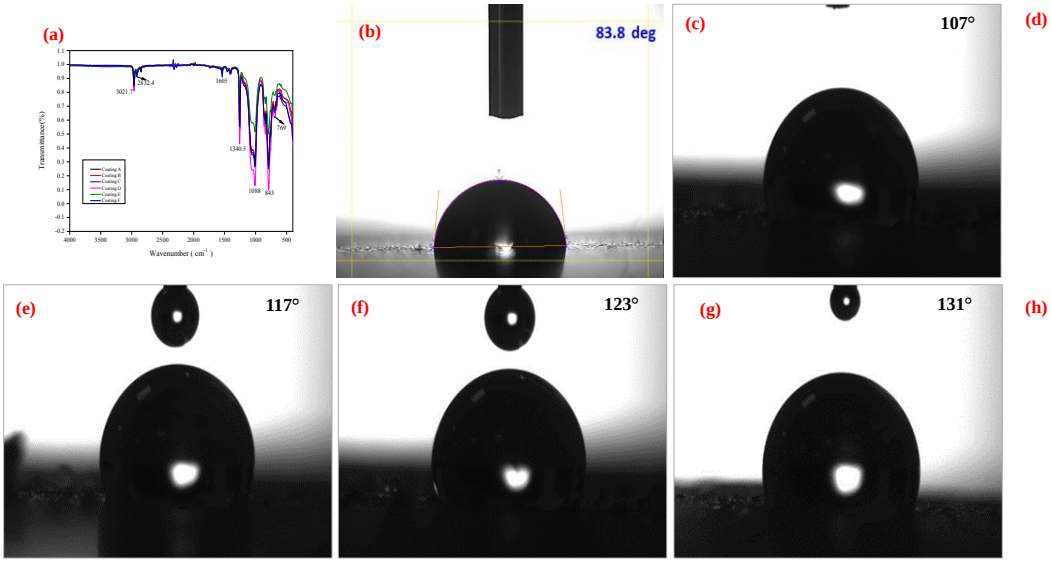

Figure 2.(a) Chemical composition, and (b)-(h) Contact angle of water with bare (GI) and the coated surfaces-A to F.

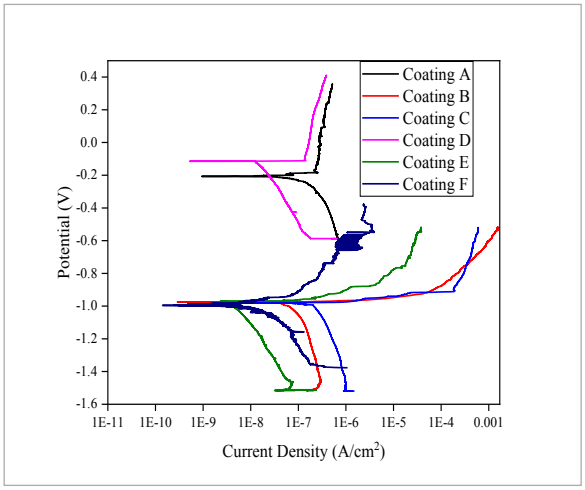

Figure 3. Potentiodynamic polarization curve of coated surfaces-A-F.