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Corrosion Behaviours of Carbon Steels Coated by Graphene Epoxy in Different Solutions

Hafid Suharyadi[1]*, Kasturi[1], Susilo Handoko[1], Burhanudin Hasan[1], Daniswara Hasta Pratama[1]

[1] *Department of Refinery Mechanical Engineering, Politeknik Energi dan Mineral Akamigas, Ministry of Energy and Mineral Resources, Jl. Gajah Mada 38, Cepu 58315, INDONESIA*

*Corresponding Author: hafid.suharyadi@esdm.go.id
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Abstract

Corrosion of carbon steel pipelines is a significant challenge in industrial applications, particularly in acidic and saline environments. This study investigates the corrosion resistance of ASTM A53 Grade B carbon steel coated with a graphene-epoxy composite. A 2%-wt graphene-epoxy coating was applied to the substrates using the bath method. Corrosion performance was assessed through potentiodynamic polarization in CH_3COOH 0.1 M, H_2SO_4 0.1 M, HCl 0.1 M, and NaCl 3.5% solutions at room temperature. Scanning Electron Microscopy (SEM) analysis provided insights into coating thickness and elemental distribution. Results indicate significant improvements in corrosion resistance, with inhibition efficiencies exceeding 97% in HCl and H_2SO_4 solutions. A notable reduction in corrosion rate and current density was observed across all coated samples, with the graphene-epoxy layer forming a robust barrier against aggressive ions. SEM analysis revealed uniform graphene dispersion within the epoxy matrix and a consistent coating thickness of ±149.9 µm, supporting the enhanced corrosion resistance. However, the coatings exhibited reduced efficacy in CH_3COOH, attributed to potential degradation of the epoxy matrix in organic acid conditions. These results demonstrate the potential of graphene-epoxy coatings as potent anticorrosion agents for a variety of industrial applications, especially those that are acidic and chloride-rich. Future research should focus on optimizing formulations for organic acids to expand the applicability of this technology.

1. Introduction

Pipeline corrosion drastically shortens their lifespan and raises maintenance expenses, especially in acidic areas. To overcome these obstacles, effective coatings are required [1–3]. Operators in the oil and gas industries, petrochemicals, and power plants are growing increasingly concerned with corrosion management planning throughout the production process as the expense of pipe corrosion control in the sector rises [4-5]. Design data for new oil and gas fields is including corrosion information from field data that is now available [6]. The goal is to create corrosion approaches that will improve production and increase pipeline design life.

Utilizing coatings as inhibitors to mitigate corrosion on metal surfaces proves useful due to its affordability, functionality, and rapid application [7-8]. These days, it's common practice to employ green inhibitors which come from renewable sources [9,10]. The effectiveness of organic compounds as inhibitors has also been studied in a few earlier studies [11–13]. Green inhibitors' toxicity, poor thermal stability in acidic environments, and low effectiveness at high doses, however, limit their applications [12-14].

There have been evaluations of using graphene to prevent corrosion [15–17]. High surface area, impermeability, and electrical conductivity of graphene are the primary drivers of its use [18]. Additionally, graphene has been functionalized using organic functional groups to improve its conductivity, corrosion resistance, and dispersibility in organic liquids [19]. The focus of our work is on the utilization of epoxy due to its excellent properties such as the interfacial binding, tensile strength, and barrier to corrosion [20]. In addition, epoxy is reasonably priced and non-toxic [21]. Although the usage of graphene coatings in various corrosive conditions has been investigated, more research is necessary before they may be used in conjunction with epoxy resin.

This study builds on previous research by utilizing the most recent composition of graphene-epoxy composite and testing its performance in various corrosive solutions. The substrates of ASTM A53 Grade B carbon steels are produced. We use corrosion potentials, Tafel slopes, current densities, and corrosion rates to figure out how well the graphene epoxy stops corrosion. In addition, the inhibition efficiency is calculated to measure the degree to which graphene-epoxy coatings are effective in preventing corrosion on carbon steel in the corrosive solutions. The components of graphene-epoxy coatings can also be mapped using scanning electron microscopy.

Nomenclature

C	corrosion rate, mmpy
EW	the equivalent weight of carbon steel
i_{corr}	the corrosion current density of coated sample, μA/cm^2
i^0_{corr}	the corrosion current density of uncoated sample, μA/cm^2
IE	the inhibition efficiency, %
k	3.27×10^{-3} mm.g/μA.cm.yr
U_c	the overall uncertainty, μm
U_{inst}	the instrument's standard uncertainty, μm
U_{repeat}	the variability observed in repeated measurements, μm
U_{env}	the uncertainty contribution from the environment, μm

Greek symbols

β_α	anodic Tafel slope, mV/dec
β_c	cathodic Tafel slope, mV/dec
ρ	the density of carbons steel, g/cm^3

Abbreviations

ASTM	American Society for Testing and Materials
SEM	Scanning electron microscope

2. Materials and Methods

This section describes the materials and methods used in this study. To start, we describe the materials: thin layer graphene nanoplatelets, epoxy, and the substrates (pipe carbon steels). The methods include substrate coatings, solution preparations, potentiodynamic polarizations, and characterizations.

2.1 Materials

Thin layer graphene nanoplatelets were a raw material in the experiment. The bulk density varied from 0.07 to 0.11 g/cm3. The graphene particles were bonded by Sigma Aldrich's Araldite 506 epoxy glue. The epoxy glue was critical in establishing a connection between the metal area and the graphene layers. The substrates were constructed using pipe carbon steels (CS) ASTM A53 Grade B Sch. 40. The substrates measured 107 mm inside, 110.4 mm outside, and 4 mm thick. For the experiment, the substrates, which had a density of 7.9 g/cm^3, were divided into 4×3 cm2 pieces. Table 1 displays the chemical compositions of the substrates.

Table 1 *The compositions of CS weight ratio (wt.%)*

Composition	C	Mn	Ph	S	Co	Ni	Cr	Mo	Va	Fe
weight ratio	0.30	1.2	0.05	0.045	0.40	0.40	0.40	0.15	0.08	Balance

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2.2 Methods

This section discusses the substrate coating using bath method, solution preparations, corrosion measurements using potentiodynamic polarization, and the characterizations using a scanning electron microscope (SEM).

2.2.1 Substrate Coatings

Initially, functionalized graphene-epoxy coatings were deposited using a bath approach to the polished substrates. The substrates were cleaned using sandpaper, ethanol, and deionized water before each surface coating. To ensure that no impurities remained, the substrates were then dried using airflow. For 30 minutes, a homogeneous solution was achieved by combining graphene nanoplates with 50 milliliters of pure ethanol and stirring. An hour of sonication was then required to produce a homogenous mixture of graphene and ethanol. After adding 25 grams of epoxy and hardener to the mixture, the graphene was dispersed throughout the epoxy matrix for an hour using sonication. The produced substrates were subsequently covered with a uniform 2%-wt-graphene-epoxy mixture by brushing it on. Following a 24-hour heating period at 60°C to eliminate the solvent, the composite coatings were dried for ninety minutes at 100°C. After coating, samples were left to rest for six hours at ambient temperature.

2.2.2 Solution Preparations

Test solutions were prepared by diluting concentrated solutions to CH_3COOH 0.1 M, H_2SO_4 0.1 M, HCl 0.1 M, and NaCl 3.5% particularly. CH_3COOH represents weak acidic conditions to mimic environments with organic acid presence. In industrial applications, extremely harsh conditions are simulated using H_2SO_4, a strong acidic solution. HCl replicates chloride-rich conditions, which are common in chemical processes or marine habitats. Seawater and other saline-based processes are represented by NaCl. Two substrates, i.e. the uncoated and coated samples were prepared for each solution in potentiodynamic polarizations.

2.2.3 Potentiodynamic Polarizations

The ability of graphene-epoxy coatings in mitigating corrosion inside an acidic solution was carefully analyzed employing a CorrTest Potentiostat/Galvanostat (Model CS350) apparatus configured with a three-electrode cell system. The working electrodes were samples of substrates having an uncovered surface of 0.8 cm^2. A graphite rod functioned as the counter electrode, whilst the reference electrode employed was the saturated calomel electrode. At open circuit potential, the samples were initially submerged in the test fluid for 30 minutes to ensure a steady state condition. Potentiodynamic polarization was set at a scan rate of 0.50 mV/s, ranging from -0.05 V to +0.05 V. Corrosion potential (E_{corr}) and current density (i_{corr}) were calculated from Tafel plots of potential E as a function of log current I. The corrosion rate of the carbon steel sample was determined as follows [22]

$$C = \frac{K \times i_{corr} \times EW}{\rho} \quad (1)$$

where C is the corrosion rate (mmpy), K is 3.27×10^{-3} mm.g/μA.cm.year, i_{corr} is the current density (μA/cm^2), EW is the carbon steel equivalent weight, and ρ is the density of the carbon steel (g/cm3). The inhibition efficiency (IE) was assessed based on the current densities of both the uncoated and coated graphene samples as outlined below [23]

$$IE = \frac{i^0_{corr} - i_{corr}}{i^0_{corr}} \quad (2)$$

where i^0_{corr} is the current density of uncoated sample in (μA/cm^2), i_{corr} is the current density of coated sample in (μA/cm^2).

2.2.4 Characterizations

The cross sections of a 2-weight-percent sample coated with epoxy-graphene were analyzed using an Inspect S50 scanning electron microscope (SEM) at a magnification of 250 times and an acceleration voltage of 20 kV for elemental mapping. This approach assessed a specific region to generate a spectrum that encompasses the information regarding the elements.

2.2.5 Uncertainty Analysis

To analyze the uncertainty of thickness measurements, a method to expressing uncertainty in measurement was applied. The process involved identifying sources of uncertainty (instrument calibration, repeatability

assessment, and environmental effects) and combining these components to estimate overall uncertainty. The combined uncertainty was calculated using the root-sum-square (RSS) method as follows [24]

$$U_c = \sqrt{u_{inst} + u_{repeat} + u_{env}} \quad (3)$$

where U_c = the overall uncertainty (µm), u_{inst} = the instrument's standard uncertainty (µm), u_{repeat} = the variability observed in repeated measurements (µm), and u_{env} = the uncertainty contribution from the environment (µm). The expanded uncertainty (U) was multiplied by a coverage factor (k = 2) to estimate the expanded uncertainty, representing a 95% confidence level [24].

3. Results and Discussion

This section covers the results of corrosion rate and inhibition efficiency using potentiodynamic polarization. In addition, the elemental mapping of carbon using a scanning electron microscope (SEM) will be shown. The discussion of the results will be

3.1 Results

The following characteristics are measured for corrosion qualities using the potentiodynamic polarization method: corrosion potential, Tafel slopes, corrosion current densities, corrosion rates, and inhibition efficiency. Tafel plots of the substrates without and with the inclusion of a graphene-epoxy composite exposed under CH_3COOH 0.1 M, H_2SO_4 0.1 M, HCl 0.1 M, and NaCl 3.5% are shown in Fig. 1.

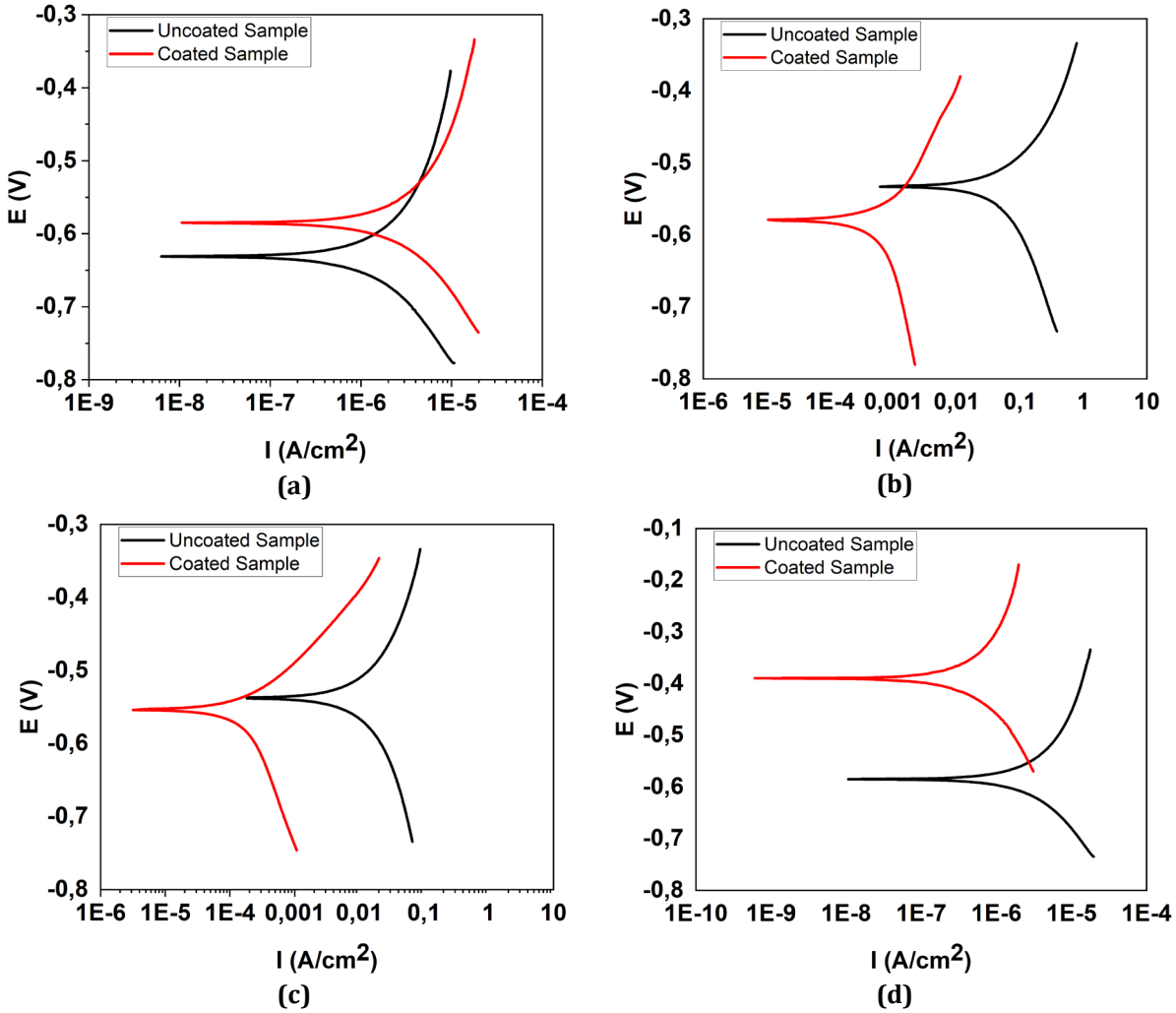

Fig. 1 *Potentiodynamic polarization curves for the uncoated and coated samples in different solutions: (a) CH3COOH; (b) H2SO4; (c) HCl; and (d) NaCl*

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In all solutions at room temperature, Table 2 displays the potentiodynamic polarization characteristics (corrosion potential, corrosion current densities, β_a and β_c slopes, corrosion rate, and inhibition efficiency) of the substrates with and without graphene epoxy coatings.

Table 2 *Potentiodynamic polarization parameters of the substrates without and with graphene epoxy coating in different solutions at room temperature*

Solutions	Coating	E_{corr} (V)	i_{corr} ($\mu A/cm^2$)	β_a (mV/dec)	β_c (mV/dec)	C (mmpy)	IE (%)
CH_3COOH	uncoated	-0.6314	4.14	566.10	317.95	0.047	
CH_3COOH	coated	-0.6322	4.77	703.50	339.98	0.054	-15.3
H_2SO_4	uncoated	-0.5331	1.22	222.32	448.15	1392.90	
H_2SO_4	coated	-0.5795	2.59	272.75	3826.30	29.50	97.9
HCl	uncoated	-0.5376	6.39	650.25	1094.20	727.48	
HCl	coated	-0.5535	2.67	103.72	358.13	3.04	99.6
NaCl	uncoated	-0.5848	8.40	656.95	334.71	0.096	
NaCl	coated	-0.3897	3.27	3714.90	665.10	0.037	61.1

The elemental mapping approach can be used to assess the distribution of carbon atoms in the graphene-epoxy layer by using SEM. Understanding the outcomes of combining epoxy with graphene using the bath approach requires knowledge of this SEM testing technique. Fig. 2 shows that the generated graphene-epoxy layer had an average thickness of 149.9 µm.

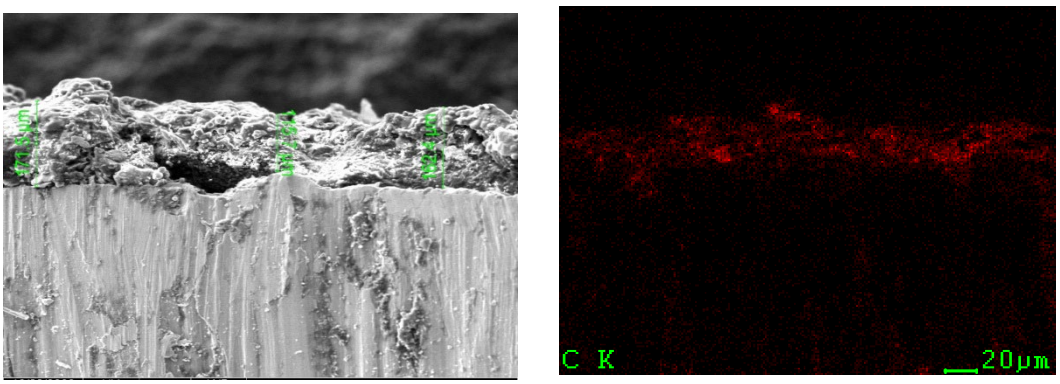

Fig. 2 *SEM images showing (left) the cross-section and (right) the elemental mapping of 2 wt% graphene coated sample*

3.2 Discussion

Graphene-epoxy coatings on the substrate give protective layers that prevent attacks from corrosive solutions. Potentiodynamic polarization studies conducted on all samples in various electrolyte solutions reveal that The incorporation of graphene-epoxy inhibitors results in an increase of corrosion potential values that is progressively positive. The corrosion potential (E_{corr}), as previously mentioned, represents a material's vulnerability to corrosion events [25, 26]. The coated samples showed a modest shift in corrosion potential in all solutions, suggesting that the graphene-epoxy coatings created a protective barrier. NaCl showed the biggest positive shift (from -0.5848 V to -0.3897 V), indicating that the coating was effective in lowering the substrate's susceptibility to corrosion in saline environments. Therefore, the graphene-epoxy inhibitor's steadily rising positive corrosion potential value demonstrates its capacity to prevent corrosion attacks on the substrate surface.

A shift from uncoated samples to coated samples is also observed in the corrosion current densities (i_{corr}). In comparison to the uncoated samples, the coated samples have a lower corrosion density value. This demonstrates how harder it is for the corrosive ions in the electrolyte solution to permeate the surface of the substrates [27, 28]. The inhibition efficiency for each sample with various electrolytes also demonstrates this trend. The inhibitory impact of the coatings was confirmed by the coated samples' consistent lower current densities compared to the uncoated ones. i_{corr} decreased from 6.39 $\mu A/cm^2$ (uncoated) to 2.67 $\mu A/cm^2$ (coated) in HCl, for example, demonstrating better performance in reducing the aggressive effect of chlorides.

The impact of the graphene-epoxy inhibitor layer on the samples is indicated by the corrosion rate (C). When a protective layer forms on the substrates, it effectively blocks ions from the acid and salt solutions on attacking

the substrates. The layer of graphene-epoxy sticks hard to the substrate surface. These outcomes agree with the inhibition efficiency values determined by analyzing the measurement data for all solutions. In H_2SO_4, the rate showed a sharp decline in acidic attack, going from 1392.90 mmpy to 29.50 mmpy. The coating's resilience in chloride-rich environments was confirmed by the reduction of NaCl from 0.096 mmpy to 0.037 mmpy. The results vary considerably with other works [29-30].

Especially in acidic environment, high inhibition efficiency (*IE*) was noted. In HCl, achieved 99.6% efficiency indicates the coating can successfully combat hostile chloride ions. In H_2SO_4, achieved 97.9% shows comparable protective qualities. The anomalous negative efficiency (-15.3%) in CH_3COOH may be the result of epoxy breakdown in acetic acid or insufficient coating adherence. The reduced effectiveness in CH_3COOH could also stem from partial degradation of the epoxy matrix in organic acids. Tafel slopes for the anodic (β_a) and cathodic (β_c) states change indicating that the graphene-epoxy layer changed the electrochemical kinetics. Greater β_a shifts demonstrate that the coating slows down metal oxidation by primarily inhibiting the anodic process.

The corrosion current density (i_{corr}) and corrosion rate (C) decreased significantly in all coated samples, especially in HCl, where the highest inhibition efficiency (99.6%) was observed. This suggests that the graphene-epoxy coating effectively resists chloride ions, which are known for their aggressive corrosion potential. In NaCl, although the inhibition efficiency (61.1%) was lower compared to H_2SO_4 or HCl, it still demonstrates significant protection, indicating the coating's utility in saline environments. Other studies [31,32] also found graphene coatings to exhibit superior anticorrosion performance due to their impermeability and hydrophobicity. Similarly, the observed performance in acidic environments aligns with findings [28-30] who reported graphene's effectiveness in chloride-rich acidic solutions.

SEM examinations reveal that the sample coated with the graphene-epoxy inhibitor has an average layer thickness of ±149.9 µm in its cross-section (refer to Fig. 2). This thickness is adequate to shield the substrate surface from corrosion. Additionally, the uniform distribution of graphene, or C atoms, in the graphene-epoxy inhibitor layer has been observed. This demonstrates the effectiveness of the bath method approach in mixing graphene and epoxy solutions uniformly. Consequently, the graphene-epoxy inhibitor layer creates a three-dimensional network that effectively blocks the attack of corrosion agent ions on the corrosion process [20-28]. The expanded uncertainty of ±0.77 µm across all measurements indicates consistent measurement accuracy within the established confidence level. The uniformity of uncertainty across different areas validates the reliability of the measurement system under controlled environmental conditions.

4. Conclusions

A study was carried out to investigate the corrosion behaviors of graphene-epoxy coatings on carbon steel substrates immersed in CH_3COOH 0.1 M, H_2SO_4 0.1 M, HCl 0.1 M, and NaCl 3.5%. Graphene-epoxy coatings significantly reduce corrosion rates and current densities, with inhibition efficiencies exceeding 97% in HCl and H_2SO_4 solutions. SEM analysis confirms uniform coating thickness (±149.9 µm) and effective graphene dispersion, contributing to superior anticorrosion performance. The coating's performance varies with solution type, excelling in chloride and acidic environments while requiring optimization for organic acids like CH_3COOH. These findings position graphene-epoxy coatings as a viable anticorrosion solution for diverse industrial applications, with potential for further development to address specific environmental challenges.

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Conflict of Interest

The authors affirm that there are no conflicts of interest corresponding to the publication of this paper.

Author Contribution

The contributors have outlined their respective roles in this paper as follows: ***development and planning of the study:*** *Hafid Suharyadi and Kasturi;* ***data collection:*** *Burhanudin Hasan and Daniswara Hasta Pratama;* ***analysis and interpretation of results:*** *Hafid Suharyadi, Susilo Handoko, and Kasturi;* ***draft manuscript preparation:*** *Hafid Suharyadi and Burhanudin Hasan. All contributors examined the findings and accepted to the final iteration of the document.*

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