Electrodeposition of Cu-Reinforced Polyaniline Coating for Protection of AH36 Steel in Natural Seawater

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Abstract: The marine environment is highly corrosive for mild and low alloy steels. This study aimed to enhance the corrosion resistance of the AH36 steel in a saline medium by coating it with a copper particles reinforced polyaniline (PANI) layer. PANI and Cu particles were grown on the steel surface by electrodeposition methods. Firstly, PANI was electropolymerized in the presence of oxalic acid, followed by the electrodeposition of Cu particles at different deposition times. The coating showed a well-distribution of Cu particles in the polymer matrix and excellent adhesion. Furthermore, the Cu particles and PANI-coated steels exhibited corrosion resistance significantly in the saline medium compared to the bare substrate and pristine PANI-coated samples. The improved corrosion protection of a Cu@PANI coating on the AH36 steel could contribute to forming a physical barrier by filling Cu particles on the PANI pores.

Keywords: electrodeposition; marine corrosion; AH36 steel; polyaniline; copper particles

1. Introduction

AH36 steel substrate has been widely used as the substrate materials for marine platforms such as ship hulls, off-shore structures, steel bridges, sheet piling, and harbor-side facilities [1-3]. However, it is highly corrosive due to prolonged exposure to the seawater environment, requiring the means to protect steel against corrosion. So far, the coating and cathodic protection have been considered effective leading solutions for corrosion resistance of the steel substrates [4–6]. Among the coating materials, polyaniline (PANI) has been considered a potential candidate for steel corrosion protection because it forms a physical barrier against aggressive chemical agents [7–9]. In addition, PANI has environmental stability, low-cost production, and relative conductivity [10,11]. Besides, PANI possesses abundant polar groups that may act as polymeric inhibitors and shift the potential of the coated substrate to a value where the underlying metal’s corrosion kinetics are lowered [7]. For example, Camalet et al. [12] reported that the PANI films on the mild steel prepared by electrodeposition in an oxalic acid medium had good mechanical and enhanced anticorrosion in an acidic solution. However, the PANI coating typically formed a porous structure on the surface of the substrate materials, resulting in accessing the corrosive reagents and decreasing the anticorrosive property.

Metal deposition in the PANI matrix was conducted to reduce the porous structure of the polymer coating [13–17]. As a result, the metal/polymer films had improved conductivity and were suitable for various applications, including electrochemical sensors and electrocatalytic oxidation. Recently, Jiang et al. [7] prepared PANI/Ni(OH)2 using an electrodeposition technique and investigated it for corrosion protection of 304 stainless steel. The coating showed excellent anticorrosion ability in 3.5% NaCl solution because it
was more compact with fewer pores on the PANI/Ni(OH)\textsubscript{2} surface. However, the corrosion protection of the coated 304SS samples was tested in the laboratory using NaCl solution, not in a seawater medium.

Copper is a passive metal well-known for seawater applications due to its corrosion resistance and anti-fouling properties \cite{18–21}. Moreover, it is reported that the Cu coatings on steel with good adhesion and uniform surface coverage could be easily made and scalable using electrodeposition \cite{18,22}. Besides, the corrosion of copper in marine media forms a film on the surface, which generally reduces the rate of both the anodic and cathodic charge transfer process \cite{18}.

In this study, a Cu + PANI coating was electrodeposited onto the surface of AH36 structural steel. The coatings were characterized by FTIR, SEM, and XRD techniques. In addition, the anticorrosion ability of the coated substrate materials was evaluated in natural seawater.

2. Materials and Methods

2.1. Materials

Oxalic acid (H\textsubscript{2}C\textsubscript{2}O\textsubscript{4}.2H\textsubscript{2}O, 98%), aniline (C\textsubscript{6}H\textsubscript{5}NH\textsubscript{2}, 99%), sodium hydroxide (NaOH, ≥97%), copper sulfate pentahydrate (CuSO\textsubscript{4}.5H\textsubscript{2}O, ≥98%), ethylenediaminetetraacetic acid disodium salt hydrate (C\textsubscript{10}H\textsubscript{14}N\textsubscript{2}O\textsubscript{8}Na\textsubscript{2}.2H\textsubscript{2}O, ≥95%), hexamethylenetetramine (C\textsubscript{6}H\textsubscript{12}N\textsubscript{4}, ≥99%), and hydrochloric acid (HCl, 37%) were purchased from Sigma-Aldrich (armstadt, Germany) and used as received. Other reagents were of analytical grade and used as received. Water was distilled in the laboratory.

The AH36 structural steel (80 mm × 10 mm × 3 mm) was used as samples with the following compositions in wt%: C ≤ 0.18; Si ≤ 0.5; Mn ≤ 0.9; Cr ≤ 0.2; Cu ≤ 0.05; Ni ≤ 0.4; Mo ≤ 0.08; P ≤ 0.035; S ≤ 0.035; Nb ≤ 0.02; Ti ≤ 0.02; Al ≤ 0.015, and balance Fe. First, the AH36 steel sheets were polished with SiC paper and ultrasonically cleaned in acetone and distilled water several times. Then, the samples were immersed in an aqueous solution consisting of 0.35 wt% hexamethylenetetramine and 0.5 M HCl for 30 min and then cleaned with distilled water and dried at room temperature overnight.

2.2. Electrodeposition of PANI and Cu on the AH36 Steel

The electrochemical deposition of the PANI film and Cu particles and electrochemical corrosion behavior was conducted using a Metrohm PGSTAT 204N electrochemical device (Metrohm Autolab BV, Utrecht, The Netherlands) at room temperature. The electrochemical cell consists of three electrodes, an Ag/AgCl reference electrode, a platinum counter electrode, and an AH36 plate (5 cm × 5 cm) as a working electrode. The exposed area was 3.46 cm\textsuperscript{2}.

Polyaniline (PANI) was electropolymerized onto AH36 steel in an aqueous solution of 0.5 M oxalic acid containing 0.1 M anilines. The electrodeposition process was conducted by cyclic voltammetry (CV) under a potential window of −0.5–1.6 V at a scan rate of 10 mV/s with 10 scans. The PANI@AH36 was washed with distilled water and dried at 40 °C overnight.

The electrochemical deposition of Cu particles onto the AH36 steel and PANI@AH36 steel was performed using chronoamperometry (CA) in a solution containing 0.3 M NaOH, 0.15 M EDTA, and 0.02 M CuSO\textsubscript{4} at pH 8 and a constant applied potential of −1.1 V. The Cu@AH36 sample was deposited for 360 s. In the cases of the Cu/PANI@AH36 samples. The deposition times were varied for 180, 360, and 720 s, which were named Cu/PANI@AH36-1, Cu/PANI@AH36-2, and Cu/PANI@AH36-3. After the deposition process, the coated samples were cleaned with distilled water, dried at 40 °C overnight, and used for characterization and anticorrosion investigation.
2.3. Characterization and Electrochemical Corrosion Test

The coating was shaved off and characterized by Fourier-transform infrared spectroscopy (FTIR, Nicolet iS10, Thermo Scientific, Waltham, MA, USA) within a range of 500–4000 cm⁻¹ at a resolution of 16 cm⁻¹ within 32 scans. The surface morphology and crystallinity of the Cu@PANI@AH36 steel were determined using a scanning electron microscope (SEM, S-4800, Hitachi, Tokyo, Japan) and X-ray diffraction (XRD, PANalytical, X’Pert-PRO MPD, Almelo, The Netherlands) using Cu Kα radiation, respectively. The adhesion strength of the coatings was determined according to ASTM D4541 by a PosiTest AT pull-off adhesion tester (PosiTest AT, DeFelsoko, Ogdensburg, NY, USA) with 20 mm dollies. All tests were repeated three times to ensure the repeatability of the measurements. Coating thickness was measured by Defelsko Positector 6000; each sample was measured in 5 positions (four in four corners with 1 cm from the edges, one in the middle), and the average value was reported. The electrochemical performance and corrosion protection of the uncoated and coated samples were evaluated in 3.5 wt% NaCl solution and natural seawater medium.

3. Results and Discussions

3.1. Characteristics of the Coated Samples

Scheme 1 shows the electrodeposition processes with some digital photos to form a compact coating onto the AH36 structural steel regarding marine corrosion protection. Firstly, the PANI was coated onto the cleaned AH36 steel using an electropolymerization method in an oxalic acid solution. The formation of a dark blue film on the steel substrate was observed, indicating the deposition of PANI. After that, the electrodeposition of Cu particles on the PANI—coated steel was conducted in the EDTA + CuSO₄ solution. The coating color was changed from dark blue to dark purple according to the presence of red Cu particles. These particles reduce the PANI coatings’ porosity and improve the adhesion between the coating and substrate and uniform surface coverage.

![Scheme 1](image)

Scheme 1. The schematic diagram for PANI and Cu electrodeposition on the AH36 substrate and the proposed corrosion protection mechanism.

Figure 1 presents the surface morphology of the bare AH36 (Figure 1a,b), cleaned AH36 (Figure 1c,d), and Cu particles-coated cleaned AH36 (Figure 1e,f) at different magnifications. Many particles on the surface of the bare steel (Figure 1a,b) were mostly removed by a washing step. It was confirmed by observing the cleaned substrate (Figure
This treatment aimed to provide a better surface for the growth of PANI, resulting in improving the adhesion of the polymer coating. Besides, the cleaned AH36 surface was coated with many Cu particles (Figure 1e,f). However, several pores on the substrate surface may increase the corrosive agents’ penetration into the AH36 steel. Therefore, it suggests coating the AH36 surface with PANI and Cu nanoparticles.

Figure 1. SEM images of (a,b) bare AH36, (c,d) cleaned AH36, and (e,f) Cu@AH36 at different magnifications.

Figure 2 shows the surface morphology of pristine PANI coating and various PANI + Cu coatings on the cleaned AH36 structural substrates at different magnifications (insets are high-magnification images). The pristine PANI coating presented a highly porous structure with large pores (Figure 2a). However, these pores were filled almost entirely with copper particles (Figure 2b–d). The compact structures of the PANI + Cu layers were observed at low- and high-magnification SEM images. These as-formed particles with several hundred nanometers were occupied in the PANI pores, reducing porous structures with abundant open space and preventing the mass within the substrate surface. Therefore, the anticorrosion of the hybrid coating could be enhanced. However, the Cu/PANI@AH36-1 sample prepared with a short deposition time (Figure 2c) shows several pores on the surface, which may also promote the transport of corrosive ions or agents.

In contrast, the Cu particles became bigger and not well-distributed on the surface of the substrate when the longer deposition time was applied, as seen in Figure 2d. As a result, the thickness of the coatings was increased, as shown in Table 1. The results showed that the PANI + Cu coating is thicker than the pure PANI and bare Cu coatings, suggesting the presence of Cu particles on both surfaces and inside the PANI layer. Besides, the adhesion of the coatings was slightly increased according to the increase in the deposition time (Table 1). Therefore, the Cu/PANI@AH36-3 sample was expected to have the highest anticorrosion capacity and was chosen for EDS and FTIR analysis.
Table 1. The thickness and adhesion of the different coatings.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Thickness (µm)</th>
<th>Adhesion (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PANI@AH36</td>
<td>47.2 ± 1.5</td>
<td>3.89 ± 0.01</td>
</tr>
<tr>
<td>Cu@AH36</td>
<td>22.8 ± 0.7</td>
<td>5.03 ± 0.01</td>
</tr>
<tr>
<td>Cu@PANI-1</td>
<td>52.6 ± 1.1</td>
<td>4.04 ± 0.01</td>
</tr>
<tr>
<td>Cu@PANI-2</td>
<td>52.9 ± 1.6</td>
<td>4.79 ± 0.01</td>
</tr>
<tr>
<td>Cu@PANI-3</td>
<td>53.4 ± 0.5</td>
<td>4.93 ± 0.01</td>
</tr>
</tbody>
</table>

Figure 2. SEM images of (a) PANI@AH36, (b) Cu/PANI@AH36-1, (c) Cu/PANI@AH36-2, and (d) Cu/PANI@AH36-3.

Figure 3 presents the XRD profiles of bare and cleaned AH36 substrate, PANI@AH36, Cu@AH36, and Cu/PANI@AH36 samples. For the bare substrate (Figure 3a), there are several peaks at around 20 of 20 and 32°, which may be attributed to ferric oxides. After cleaning, these peaks disappeared. There is a crystal diffraction peak at 20 of 44.3° for Fe (JCPDS file No. 00-006-0696) in the AH36 (Figure 3b). For the PANI@AH36 sample (Figure 3c), the intensity of the prominent characteristic peak of Fe was reduced, indicating that a PANI layer was coated on the surface of the substrate. In the XRD pattern of the Cu@AH36 sample (Figure 3d), three characteristic peaks for copper (JCPDS file No. 03-065-9743) were observed at 20 of 44.8, 56.7, and 64.9° according to the (111), (200) and (220) planes. After the deposition of Cu particles on the PANI-coated substrate at different reaction times, all characteristic peaks of copper were observed. The peaks’ intensities were slightly increased, possibly due to the increase of the Cu coating layers (Figure 3e–g). The results are in agreement with the observations in the SEM images.
Figure 3. XRD patterns of the (a) bare AH36, (b) cleaned AH36, (c) PANI@AH36, (d) Cu@AH36, (e) Cu/PANI@AH36-1, (f) Cu/PANI@AH36-2, and (g) Cu/PANI@AH36-3.

Figure 4 shows the EDS spectrum of the Cu/PANI@AH36-3 surface. The insets are the taking position and chemical composition. Three main elements, C, Fe, and Cu, were detected on the coated steel’s surface, indicating the presence of PANI and Cu particles. Fe is according to the AH36 substrate. The weight percentage of Cu is higher than that of C, suggesting that Cu particles covered the porous structure of the PANI.

![Figure 4](image-url)

Figure 4. EDS spectrum of the Cu/PANI@AH36-3 (the inset is the elemental composition of a specific area).

The chemical structure information of coated steels was determined using FTIR spectroscopy and shown in Figure 5. The spectrum of the PANI-coated sample (Figure 5a) displays the characteristic peaks at 1594 and 1505 cm\(^{-1}\) (C=C stretching of the quinoid ring and benzenoid ring) and at 1304 and 1145 cm\(^{-1}\) (C-N stretching of the secondary aromatic amine). Two peaks at 1234 and 805 cm\(^{-1}\) were ascribed to C-H stretching. The broad peak at 3442 cm\(^{-1}\) is attributed to the O-H stretching. Figure 5b shows the FT-IR spectrum of the Cu/PANI@AH36-2 sample with the presence of all peaks for PANI.
However, some peaks slightly shifted from 1594 to 1564 cm\(^{-1}\) (C=C stretching of the quinoid ring), from 1505 to 1492 cm\(^{-1}\) (C=C stretching of the benzenoid ring), and from 805 to 822 cm\(^{-1}\) (C-H stretching). The changes may be the interactions between the Cu and -NH\(_2\)/-OH groups, suggesting a well-attachment of Cu particles in the PANI matrix.

![Figure 5. FT-IR spectra of (a) PANI@AH36 and (b) Cu/PANI@AH36-3.](image)

### 3.2. Electrochemical Performance

The electrochemical performance of the uncoated and coated AH36 was measured and presented in Figure 6 and Table 2. Figure 6a shows the open-circuit potential (OCP) curves versus time for various samples in 3.5 wt.% NaCl medium. It can be seen that the potential of AH36 is more negative (-) than the copper or PANI or Cu + PANI coated samples. This proves that the Cu + PANI samples have high anticorrosion protection. On the other hand, the coating samples' anticorrosive ability was also investigated via the corrosion potential (\(E_{\text{corr}}\)) versus the corrosion current (\(i_{\text{corr}}\)). Figure 6b presents the Tafel polarization curves of the bare AH36, PANI@AH36, Cu@AH36, and Cu/PANI @AH36-2 samples. As seen in Table 2, compared with the uncoated substrate (\(E_{\text{corr}}\) of \(-713\) mV), the \(E_{\text{corr}}\) values of the coated AH36 samples are greater, ranging from \(-620\) mV to \(-447\) mV. Besides, the Cu@AH36 shows a higher \(E_{\text{corr}}\) value than the PANI@AH36 due to the higher anticorrosive nature and more compact Cu particle layer. For the Cu/PANI@AH36-2 (\(E_{\text{corr}}\) of \(-494\) mV) and Cu/PANI@AH36-3 (\(E_{\text{corr}}\) of \(-492\) mV) samples, the potential corrosion values are enhanced in comparison to others, indicating the highly effective physical barrier of the Cu + PANI coating. It can be explained that the modification of the Cu particles reduced the PANI layer’s porous structure and improved the coating’s hydrophobicity. Thus, the Cu + PANI coating prevents the permeation of aggressive ions/agents and acts as an excellent physical barrier to prevent the corrosion of the AH36 surface [23,24]. In addition, the lower \(i_{\text{corr}}\) values indicate higher anticorrosion of the tested samples. Table 2 shows the corrosion current (\(i_{\text{corr}}\)) values obtained from the bare AH36, PANI@AH36, Cu@AH36, Cu/PANI@AH36-1, Cu/PANI@AH36-2, and Cu/PANI@AH36-3 are 17.2, 11.6, 5.7, 5.5, 4.4 and 4.3 μA cm\(^{-2}\), respectively. The reduction in \(i_{\text{corr}}\) of the AH36-coated samples was consistent with their increase in \(E_{\text{corr}}\). The results showed that Cu and PANI effectively inhibited the extent of corrosion in the Cl\(^-\) environment.
Figure 6. (a) Values of the open circuit potential (OCP) and (b) Tafel polarization curves obtained from the bare AH36, PANI@AH36, Cu@AH36, Cu/PANI@AH36–1, Cu/PANI@AH36–2, and Cu/PANI@AH36–3 steels.

Table 2. Electrochemical parameters from the polarization curves and OCP measurements.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Bare AH36</th>
<th>PANI@AH36</th>
<th>Cu@AH36</th>
<th>Cu/PANI@AH36–1</th>
<th>Cu/PANI@AH36–2</th>
<th>Cu/PANI@AH36–3</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_{corr}$ (mV vs. Ag/AgCl)</td>
<td>-713</td>
<td>-620</td>
<td>-447</td>
<td>-583</td>
<td>-494</td>
<td>-492</td>
</tr>
<tr>
<td>$i_{corr}$ (μA cm$^{-2}$)</td>
<td>17.2</td>
<td>11.6</td>
<td>5.7</td>
<td>5.5</td>
<td>4.4</td>
<td>4.3</td>
</tr>
<tr>
<td>$R_p$ (Ω)</td>
<td>362</td>
<td>551</td>
<td>697</td>
<td>726</td>
<td>905</td>
<td>912</td>
</tr>
<tr>
<td>$\beta_a$ (mV/dec)</td>
<td>393</td>
<td>199</td>
<td>115</td>
<td>75</td>
<td>64</td>
<td>63</td>
</tr>
<tr>
<td>$\beta_c$ (mV/dec)</td>
<td>-66</td>
<td>-68</td>
<td>-62</td>
<td>-69</td>
<td>-67</td>
<td>-65</td>
</tr>
</tbody>
</table>

3.3. Mechanism

The protection ability of the Cu/PANI coating on the AH36 substrate in a marine environmental medium is attributed to the anodic protection of the conductive PANI polymer and Cu particles and their barrier effect. Firstly, it is reported that the primary and initial copper corrosion in seawater media is cuprous chloride, as follows [25].

$$\text{Cu} \rightleftharpoons \text{Cu}^+ + e^- \quad (1)$$

$$\text{Cu}^+ + \text{Cl}^- \rightleftharpoons \text{CuCl} \quad (2)$$

The cuprous chloride is slightly soluble and reacted to form cuprous oxide (CuO), cupric hydroxide (Cu(OH)$_2$), atacamite (Cu$_2$(OH)$_3$Cl), or malachite (CuCO$_3$·Cu(OH)$_2$) in seawater for a long time. Therefore, it forms a passive film on the surface of Cu, which protects the underlying copper metal from corrosion.

Later, the researchers found that the cuprous chloride complexes (CuCl$_2^-$, CuCl$_3^-$, CuCl$_4^-$) and cuprous oxide (Cu$_2$O) were formed in successive chloride ions of the seawater [18,22,26,27], as described in Equations (3) and (4). However, the formation rate of cuprous oxides is lower than that of the cuprous complexes because of the buffering properties of seawater.

$$\text{Cu}^+ + 2\text{Cl}^- \rightleftharpoons \text{CuCl}_2^- + \text{Cl}^- \rightleftharpoons \text{CuCl}_3^- + \text{Cl}^- \rightleftharpoons \text{CuCl}_4^- \quad (3)$$

$$2\text{CuCl}_2^- + 2\text{OH}^- \rightleftharpoons \text{Cu}_2\text{O} + \text{H}_2\text{O} + 4\text{Cl}^- \quad (4)$$
Generally, the corrosion protection of copper in seawater is attributed to the passive solid films of Cu2O, CuCl, and Cu2(OH)3Cl. This is confirmed in Table 2 that the $E_{corr}$ value of the Cu@AH36 sample ($-447$ mV) is much higher than that of the bare AH36 ($E_{corr} \approx -713$ mV).

Similarly, conductive PANI films protect the AH36 substrate mainly via the anodic protection and physical barrier effect. The main redox reactions of PANI film on AH36 substrate in seawater are described as follows [7,28].

$$\begin{align*}
\text{Fe} & \rightleftharpoons \text{Fe}^{2+} + 2e^- \\
\text{O}_2 + 2\text{H}_2\text{O} + 4e^- & \rightleftharpoons 4\text{HO}^- \\
\text{PANI-ES} + \text{OH}^- & \rightleftharpoons \text{PANI-EB} + \text{H}_2\text{O} + \text{A}^- \\
\text{PANI-ES} + 2e^- & \rightleftharpoons \text{PANI-LE} + 2\text{A}^- \\
\text{O}_2 + \text{PANI-LE} + \text{H}_2\text{O} & \rightleftharpoons \text{PANI-ES} + \text{OH}^- 
\end{align*}$$

where ES is emeraldine salt, EB is emeraldine base, and LE is leucoemeraldine.

The passive insoluble particles are formed based on the reactions of Fe$^{2+}$ ions (at the anodic zone) and the counter A$^-$ ions (at the cathodic zone). Practically, the $E_{corr}$ value of the PANI@AH36 ($-620$ mV) is much higher than that of the bare AH36 ($E_{corr} \approx -713$ mV) (Table 2). However, the deposited PANI layer is porous, as shown in Figure 2a, which may allow the quick penetration of aggressive ions and thus destroy the coating in seawater. By coating with PANI film and Cu particles, the pores in the polymer film were filled by the Cu particles. Besides, all passive insoluble components from both PANI and Cu are contributed to the anticorrosion capacity of the coating. Overall, it is concluded that the Cu particles filled in PANI porous structure and strengthened the physical barrier to prevent the aggressive ions and agents.

Moreover, it forms a passive oxide film on the interface between the coating and the AH36 substrate. Therefore, adding Cu particles to PANI film sustains the anodic protection and enhances the corrosive protection of the prepared coating in the marine environment for a long time. As can be seen in Table 2, the anticorrosion of Cu@PANI@AH36 is much improved than the PANI@AH36 and Cu@AH36 samples. Moreover, this behavior agrees with the observations in Figures 7 and 8.

**Figure 7.** Photos of the different coated samples (a) before, (b) on-site, and (c) after exposure to seawater for 60 days.
4. Conclusions

In this study, a highly compact layer of PANI and Cu particles was successfully electrodeposited on AH36 structural steel. Furthermore, the deposition of Cu particles significantly reduced the porous structure of the pristine PANI coating, increasing the $E_{corr}$ and $R_p$ values and enhancing the corrosion protection capability of the two-component coating in natural seawater medium. These findings suggest that the PANI + Cu coating is a potential candidate for protecting the AH36 substrate employed in marine environments.

**Author Contributions:** Conceptualization, N.V.C. and N.V.H.; methodology, L.H.Q. and N.Q.Q.; formal analysis and investigation, N.V.C., L.H.Q., N.D.A., D.V.K. and N.V.H.; writing—original draft preparation, N.V.C.; writing—review and editing, N.V.H., C.N.L. and L.H.Q. All authors have read and agreed to the published version of the manuscript.

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