

PANi/PPy and PANi Films on ZnNi Alloy Coated Carbon Steel; Electrochemical Syntheses and Corrosion Performances

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Abstract: Zinc–nickel alloy coating (ZnNi) was successfully deposited on carbon steel (CS) applying current density of 47 mAcm⁻² with galvanostatic technique. The ZnNi alloy coating image showed that CS metal surface was plated with a blue-grey homogeneous layer. Polyaniline film (PANI) film was synthesized with cyclic voltammetry technique from 0.10 M aniline containing 0.20 M sodium oxalate solution on ZnNi coated carbon steel (CS/ZnNi) electrode surface. And then the synthesis of top PPy film was achieved in pyrrole monomer containing acetonitrile + tetrabutyl ammonium perchlorate medium successfully. After the top PPy synthesis, it was observed that both the PANi homopolymer film and the ZnNi coating continued to exist on the CS surface. The corrosion behaviors of uncoated and coated electrodes were investigated in 3.5% NaCl solution. For this aim, the anodic polarization plots and electrochemical impedance spectroscopy technique were used. The results showed that the top PPy homopolymer film provided an effective barrier property on CS/ZnNi/PANi electrode and a remarkable anodic protection to substrate for longer exposure time.

Keywords: Alloy plating, corrosion, conducting polymer, polypyrrole, polyaniline, ZnNi

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1. Introduction

The role and importance of technical metals in today's modern world is too great to ignore. Especially the role of steel among these metals is vital for both industries and governments. While approximately 30 million tonnes of crude steel was produced in the world in 1900, this value reached 1864 million tonnes in 2020 (International Iron and Steel Institute 1978; World Steel Association 2021). This increase not only creates the modern world, but also provides an important workforce and economic value. According to the Oxford Economics report, the share of the steel industry in annual global GDP is around 3.8%. (Oxford Economics, 2019). As can be seen from these values, a healthy metal industry is one of the building blocks of a healthy economy and modern world. However, this rapidly increasing steel usage rate also increases the damage caused by corrosion reactions, which in turn causes the metals to wear out. Corrosion is an unstoppable nature event, causing loss of life and property, serious damage to the state and industry directly or indirectly and a loss of 3.5 to 5% per annum of GNP. (Hou et al. 2017; Koch et al. 2002; Üneri 2011).

For many years, researchers in many disciplines are constantly looking for new ways to protect materials from corrosion and thereby reduce damage caused by corrosion (Kilinççeker et al. 2008; Kilinççeker et al. 2013). Alloy coating is one of the techniques used for this purpose. Especially, zinc & zinc group coatings are frequently used with various coating techniques for cathodic corrosion protection of Fe group materials due to their high corrosion resistance and mechanical strength. Electrodeposition is one of the techniques used for zinc & zinc group coatings and has become widely used in many industries with unique advantages over many other coating techniques such as independence from material shape, easy process setup and control, etc. however, electroplating zinc alone is insufficient to protect materials from corrosion in the medium-long term due to the very less noble nature of zinc. Using zinc alloys with elements in the same period with zinc is one of the most commonly used methods to solve this problem in literature. Among these alloys, especially ZnNi alloy is studied more in the literature because of its excellent resistant characteristic to corrosive factors in the atmospheric environment, machining properties, weldability etc. (Abd El-Lateef et al. 2015; Bhat et al. 2021; Chouia et al. 2021; Ozyilmaz et al. 2020;

Tozar and Karahan 2014) Considering the literature studies in recent years, it is seen that conductive polymers are of interest in the field of corrosion protection due to their unique properties such as superior anticorrosive properties, redox reactions in natural environment and catalytic activity. For this aim, polyaniline and polypyrrole among conducting polymers are used for the corrosion protection of metals in a wide range of aggressive environments (Ates 2016; Kaliyannan et al. 2020; Sokolova et al. 2017).

In this study, the surface of the carbon steel (CS) electrode was plated with a zinc-nickel alloy. The obtained CS/ZnNi electrode was coated with thin polyaniline (PANi) film in an aqueous sodium oxalate solution. After that, polypyrrole (PPy) film was coated as top coating on CS/ZnNi/PANi surface in pyrrole containing acetonitrile + tetrabutyl ammonium perchlorate (TBAP) solution medium. The corrosion performances of the uncoated and coated electrodes were evaluated by EIS and anodic polarization techniques in 3.5% NaCl solution.

2. Materials and Method

All electrochemical studies were carried out on the CH Instruments 660B model electrochemical workstation (S/N: A1420) with three electrode technique in one cell. In the experiment cell, cylindrical low carbon steel electrode (CS) which chemical composition by wt. %: 0.056% C, 0.450% Mn, 0.140% Si, 0.004% S, 0.010% P, 99.34% Fe and surface area 0.0531 cm² as working electrode. The square platinum plate (99.99%) with surface area 0.50 cm² as counter electrode and Ag/AgCl (3M KCl) electrode as reference electrode was used. Working electrode was isolated with a thick polyester block outside the surfaces which was contact with solution and connects to instrument. Shortly before use, working surface was carefully polished with 400 and 1200 grid abrasive paper, respectively, and washed with 1:1 acetone:ethanol mixture and bi-distilled water, respectively. For the alloy coating, the citrate-sulphate aqueous bath containing 0.20 M ZnSO₄, 0.20 M NiSO₄, 0.20 M Na₃C₆H₅O₇, 0.10 M (NH₄)₂SO₄ was used (Winiarski et al. 2015) at natural pH of about 5.3. CS surface was coated with approx. 5 µm ZnNi alloy with chronopotentiometry (CP) technique at 47 mA/cm² constant current density at room condition. The electropolymerization was carried out with cyclic voltammetry (CV) technique at room condition. For the PANi coating, 0.10 M freshly distilled aniline monomer was added into 0.20 M aqueous sodium oxalate solution. First, alloy coated electrode (CS/ZnNi) was anodically passivated by applying one segment between -1.50 and 1.00 V potential at a scan rate of 10 mV/s. Next, the film growth curves were taken for 333 seconds at a potential range of 0.00 and 1.00 V at a scan rate 150 mV/s without removing CS/ZnNi electrode from sodium oxalate+aniline solution. After that, PPy film was carried out on PANi coated CS/ZnNi electrode (CS/ZnNi/PANi) surface in acetonitrile+0.025 M TBAP medium containing 0.10 M freshly distilled pyrrole between -0.80 and 0.80 V at 150 mV scan rate for 533 sec. For metallic characterization of

uncoated and coated electrodes, the linear sweep voltammetry (LSV) technique was used in aqueous 0.05 M EDTA+0.50 M Na₂SO₄ mixture solution a scan rate of 5 mV/s and a potential range of -1.10 and 1.80 V to show the presence of ZnNi alloy coating on the CS electrode. Anodic polarization curves were obtained from open circuit potential (E_{ocp}) to 1.80 V at 5 mVs⁻¹ scan rate, after 240 h of immersion time in 3.5% NaCl solution. Electrochemical impedance studies were recorded at measured open circuit potential values applying 7 mV of amplitude in frequency range from 10⁵ to 10⁻³ Hz. Real and imaginary components of the EIS in the complex plane and Bode plots were analyzed with the ZView2 software to estimate the parameters of the equivalent electrical circuit. Surface images were taken with Canon EOS5D high resolution digital camera equipped with Canon EF 100mm Ultrasonic Macro Lens.

3. Results and Discussion

In this study, firstly, the surface of the CS was successfully coated with approx. 5 µm thick ZnNi alloy from citrate-sulphate bath with 47 mA × cm⁻² current density. It was observed that the surface was polarized immediately at this current density and there was no change at the potential values at a large scale in the increasing time. In addition, it was seen that the potential values of the different electrodes were similar to each other (It was not shown here).

After the metallic coating, the CS/ZnNi electrodes were quickly taken into the PANi synthesis medium. In order to obtain the homogenous PANi film, the ZnNi alloy coated CS electrode were polarized by using only anodic scan from -1.50 to 1.00 V potential range with scan rate of 10 mV/s (It was not shown here). In the second stage, the growth cycle of PANi film was taken at potential range from 0.00 to 1.00 V with 150 mV/s scan rate. Fifty segments were applied to synthesis of the PANI film.

When the voltammograms were examined, the peaks obtained for surface oxidation-passivation and monomer-oxidation were observed at potentials of about -0.10 and 0.65 V, respectively. After monomer-oxidation peak was observed, current values at ongoing potentials were decreased. All these observations showed that the surface was covered with a thin polyaniline film during on anodic passivation process. A shift of the monomer oxidation peak recorded for aniline to around 0.79 V in the film growth voltammogram was seen in Figure 1. This case proved that a conductive polymer layer were formed on the alloy coated surface during the anodic passivation (Özyılmaz, 2005). The current decreases in the continuing peaks showed that the polyaniline film grew on the surface and reduces the surface conductivity. Figure 1b showed electropolymerization voltammograms for PPy synthesis on to MS/ZnNi/PANi surface. Current increases at potentials after 0.20 V in Figure 1b corresponded to oxidation of the pyrrole monomer. The current values recorded for the monomer oxidation increased at the initial scan and then those values decreased. According to this behavior, it was observed that the surface conductivity first

increased with the polypyrrole film, and then the surface conductivity decreased due to the form transformation of the PPy film.

The photographs of the CS, CS/ZnNi, CS/ZnNi/PANi and CS/ZnNi/PANi|PPy electrodes were given in Figure 2. The surface images show that a homogeneous ZnNi alloy layer is coated on the CS electrode surface with a blue-gray

color tone. The photographs of the black PANi and PPy top coatings synthesized on CS/ZnNi surface were given in Figure 2. As can be seen from the images, the surface was homogeneously coated with PANi film and the PANi coating surface was covered with a homogeneous top PPy film.

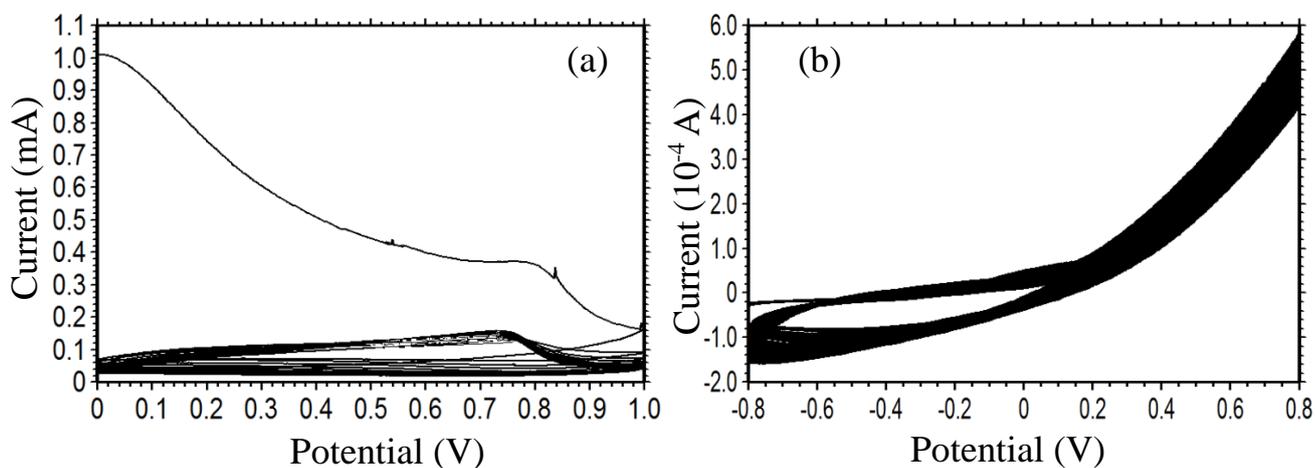


Fig. 1 The voltammograms recorded during the film growth on CS/ZnNi electrode in 0.10 M aniline containing 0.20 M sodium oxalate solution, scan rate: 10 mV/s (a) and on CS/ZnNi/PANi electrode in acetonitrile+0.025 M TBAP containing 0.10 M pyrrole, scan rate: 150 mV s⁻¹(b).

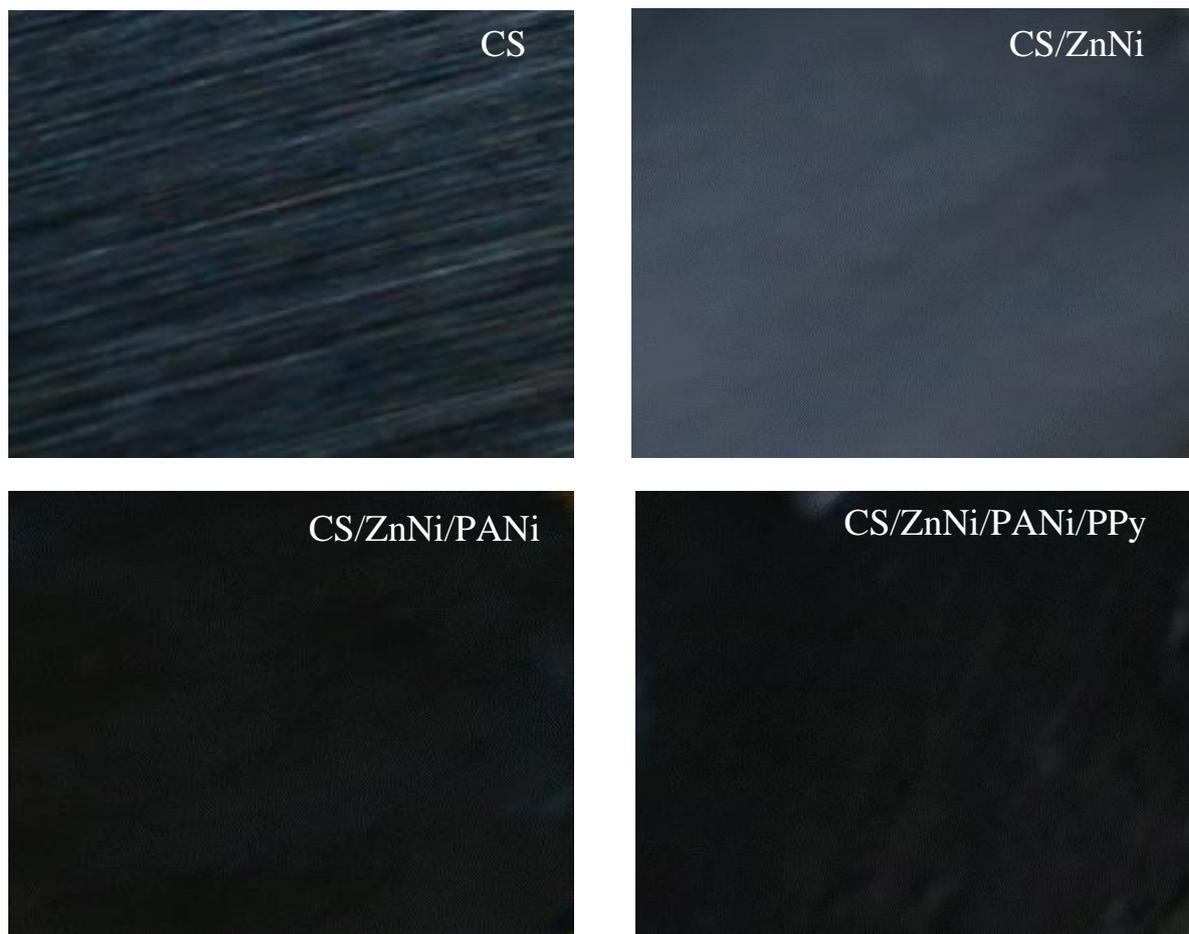


Fig. 2 The photographs of the CS, CS/ZnNi, CS/ZnNi/PANi and CS/ZnNi/PANi|PPy electrodes

Figure 3 shows metallic characterization voltammograms of the CS, CS/ZnNi, CS/ZnNi/PANi and CS/ZnNi/PANi/PPy electrodes. In Figure 3, current increases related to the primary components of the alloy were observed at the CS/ZnNi, CS/ZnNi/PANi and CS/ZnNi/PANi/PPy, unlike CS. For polymer film coated electrodes, these current increases indicated that under polymer films it was a metallic structure that can form complex with EDTA. The current peaks recorded for polymer film coated CS/ZnNi electrodes on the similar potential values of about -0.30 V, which were not recorded for bare CS clearly indicated that the polymer films were synthesized on the ZnNi alloy coated surface. When the current values on the potential values of about 0.10 V obtained for the uncoated and coated electrodes are examined, it is seen that the current values of coated electrodes were lower than the CS electrode. Accordingly, these results showed that the alloy and polymer films formed on the surface created an effective physical barrier and protect the CS electrode from corrosive environment. Besides, the lowest current value recorded for CS/ZnNi/PANi/PPy electrode indicated the presence of effective layers on the CS electrode surface (Akdağ and Özyılmaz 2017).

Anodic polarization results of the CS, CS/ZnNi, CS/ZnNi/PANi and CS/ZnNi/PANi/PPy electrodes after 240h immersion time in 3.5 % NaCl solution are given in Figure 4. The corrosion potential (E_{corr}) values obtained for CS, CS/ZnNi, CS/ZnNi/PANi and CS/ZnNi/PANi/PPy electrodes was found to be -655 mV, -611 mV, -621 mV and -516 mV, respectively. The E_{corr} values of coated electrodes shifted in the anodic region due to anodic protection than that of uncoated electrode. Besides, the most positive E_{corr} value recorded for CS/ZnNi/PANi/PPy electrode, it is easily say that the top PPy film exhibited the most effective physical barrier properties against corrosion products in the corrosive environment when compared with single ZnNi and ZnNi/PANi coatings. This occasion supported the idea that current values of CS/ZnNi/PANi/PPy electrode were significantly lowest when compared with PANi homopolymer film coated ZnNi plated carbon electrode as well as ZnNi plated carbon steel and bare carbon steel electrode. This case showed that PPy film synthesized on CS/ZnNi/PANi electrode had lowest permeability against the attack of corrosive products like dissolved oxygen and chloride ions. These anodic polarization results revealed that top PPy homopolymer film provided important corrosion protection to uncoated and coated CS electrode.

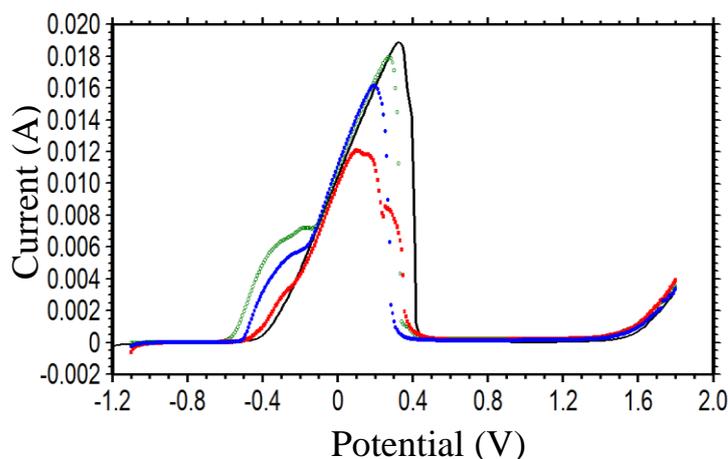


Fig. 3 Linear Sweep voltammetry recorded for MS (-), MS/ZnNi (o), MS/ZnNi/PANi (•) and MS/ZnNi/PANi/PPy (■) electrodes in 0.05 M EDTA containing 0.50 M sodium sulphate solution.

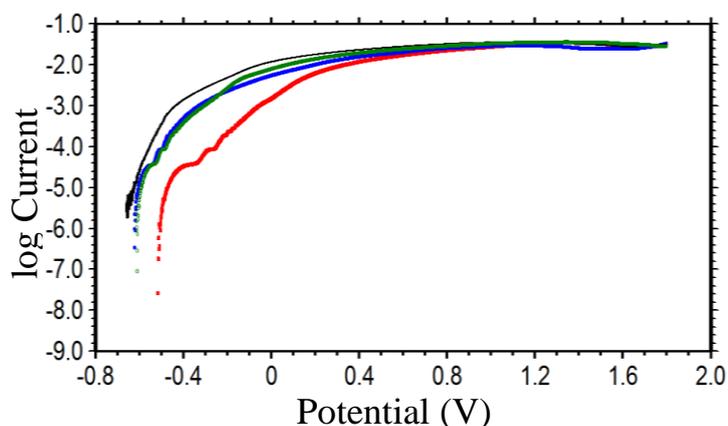


Fig. 4 Anodic polarization results recorded for MS (-), MS/ZnNi (o), MS/ZnNi/PANi (•) and MS/ZnNi/PANi/PPy (■) electrodes after 240 h of immersion time in 3.5% NaCl solution.

Figure 5 shows the Nyquist plots recorded for CS, CS/ZnNi, CS/ZnNi/PANi and CS/ZnNi/PANi|PPy electrodes after 72 and 240 h immersion times in 3.5% NaCl solution. After 72 h of exposure time, the E_{ocp} values of CS/ZnNi, CS/ZnNi/PANi and CS/ZnNi/PANi|PPy electrodes were found to be approx. -0.595, -0.599 and -0.576 V, respectively while that of bare CS electrode was -0.669 V. For the CS/ZnNi electrode, this value was significantly in the anodic direction compared to the CS electrode, despite the active zinc metal in the alloy. This behavior was the result of a stable oxide layer formed on the ZnNi alloy coating surface. On the other hand, appearance of more positive E_{ocp} value for CS/ZnNi/PANi|PPy electrode indicated prominent differences between dissolution behavior of ZnNi alloy plating and the corrosion reactions at the CS surface. This showed that PPy homopolymer film on CS/ZnNi/PANi surface exhibited an important passive film behavior against to corrosive products on carbon steel electrode. In Fig. 5(a), there were two depressed semicircles which could not be well resolved from each other from high and low frequency region, for CS/ZnNi, CS/ZnNi/PANi and CS/ZnNi/PANi|PPy electrodes, while the plot of bare CS electrode consisted of one depressed semicircle. The depressed semicircle obtained for bare CS electrode was equal to the polarization resistance (R_p) that including the total of the charge transfer resistance (R_{ct}) that is responsible for the anodic dissolution of bare metal and diffusion resistance (R_d), after 72 h of exposure time. Besides, there were R_{ct} at high frequency region and oxide layer resistance (R_o) + alloy plating resistance (R_{ZnNi}) at low frequency region for CS/ZnNi electrode, while the two depressed semicircles for CS/ZnNi/PANi and CS/ZnNi/PANi|PPy electrodes were equal to R_p including the total of $R_{ct} + R_{ZnNi} + R_o +$ polymer film resistance (R_{pf}) ranging from high to low frequency region. The corrosion current (I_{corr}) and the protection efficiency values (E%) of PANi|PPy and PANi coatings on CS/ZnNi electrode were calculated as in our earlier work (Ozyilmaz et. al. 2006). The parameters calculated for CS, CS/ZnNi, CS/ZnNi/PANi and CS/ZnNi/PANi|PPy electrodes are presented in Table 1. In Table 1, R_p values, which were analyzed with the ZView2 software, were obtained from Stern-Geary equation for calculation of corrosion current (I_{corr}) using following equation (1). B value was equal to $(\beta_a \times \beta_c) / ((\beta_a + \beta_c) \times 2.303)$; β_c and β_a were cathodic and anodic Tafel slopes, respectively. The value of B was

taken as 0.052 V for coated electrodes and 0.026 V for uncoated electrode.

$$I_{corr} = \frac{B}{R_p} \quad (1)$$

The protection efficiency values (E %) were calculated by using the following equation.

$$E\% = \frac{R_{p(uncoated)}^{-1} - R_{p(coated)}^{-1}}{R_{p(uncoated)}^{-1}} \times 100 \quad (2)$$

The R_p values obtained for CS/ZnNi/PANi|PPy electrode was found to be highest when compared with those of CS, CS/ZnNi and CS/ZnNi/PANi electrodes. Besides, I_{corr} value obtained for CS/ZnNi/PANi|PPy electrode was found to be 1.45 μ A that the lowest value when compared with those of all electrodes. This case showed the barrier effect of PPy film as top coating, after 72h of immersion time in 3.5% NaCl solution. After 240 hours, the R_p values of the CS, CS/ZnNi/PANi and CS/ZnNi/PANi|PPy electrodes were lower than the values obtained after 72 hours of exposure, while the value of the CS/ZnNi electrode was higher. These results showed that the stable oxide layers formed on the ZnNi alloy coating surface over time. The R_p value increase of CS/ZnNi electrode was observed due to the formation of stable NiO and complex $ZnO \cdot H_2O$ on ZnNi plating surface. Nevertheless, the R_p values recorded for CS/ZnNi CS/ZnNi/PANi and CS/ZnNi/PANi|PPy electrodes were found to be higher when compared with those of uncoated CS metal, after 240 h of exposure time, while the highest R_p value was obtained for CS/ZnNi/PANi|PPy electrode. In addition, the E% value of CS/ZnNi/PANi|PPy electrode was 75.36% that it was relatively higher value than those of 17.92% for CS/ZnNi and 9.87% for CS/ZnNi/PANi electrodes. On the other hand, a decrease in the I_{corr} value of the CS/ZnNi/PANi|PPy electrode was observed after 240 hours compared to the 48 hour immersion times. After 240 hours of exposure, the decrease in R_p for the CS/ZnNi/PANi|PPy electrode was indicative of a small increase in the amount of electrolyte solution in the alloy coating and the homopolymer film surfaces. Nevertheless, I_{corr} value obtained for CS/ZnNi/PANi|PPy electrode was obtained as 1.52 μ A that the lowest value than those of 3.49 for CS/ZnNi and 3.83 for CS/ZnNi/PANi electrodes. It was clear that PPy film exhibited an effective barrier property against corrosion products, whereby providing a decrease in diffusion rate of ions.

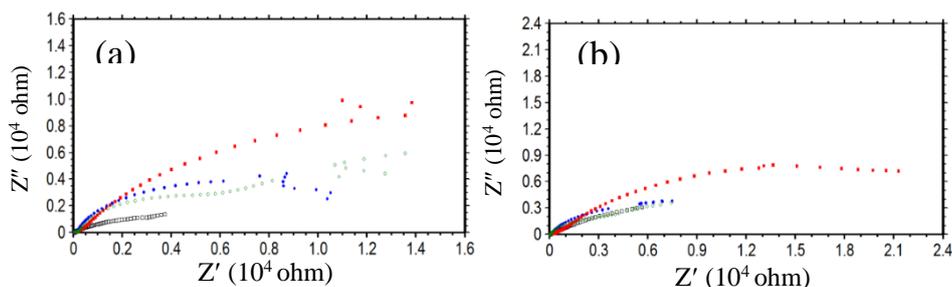


Fig. 5 The Nyquist plots recorded for MS (-), MS/ZnNi (o), MS/ZnNi/PANi (•) and MS/ZnNi/PANi/PPy (■) electrodes after 72h(a) and 240h(b) of immersion time in 3.5% NaCl solution.

Table 1. The E_{ocp} , R_p , I_{corr} and E% values recorded for CS, CS/ZnNi and CS/ZnNi/PANi and CS/ZnNi/PANi/PPy electrodes after various exposure times in 3.5% NaCl.

Electrode	t (h)	E_{ocp} (mV)	R_p (Ω)	I_{corr} (μ A)	E%
CS	72	-648	9020	-	-
	240	-665	12232	-	-
CS/ZnNi	72	-595	14148	3.67	36.25
	240	-613	14903	3.49	17.92
CS/ZnNi/PANi	72	-599	13433	3.81	32.85
	240	-616	13571	3.83	9.87
CS/ZnNi/PANi/PPy	72	-576	35813	1.45	74.81
	240	-545	34259	1.52	64.30

4. Conclusions

As a result of the study, the CS surface can be successfully coated with ZnNi alloy from the citrate sulphate bath at 47 mA/cm² current density via galvanostatic technique. It was seen from the photograph that a homogeneous ZnNi alloy coating in blue-gray color was obtained. Then, PANi coating was successfully synthesized on CS/ZnNi electrode from neutral medium. It was found that the polarization of CS/ZnNi surface was necessary for homogenous PANi film synthesis prior to monomer oxidation and film growth. And then, PPy film was carried out on CS/ZnNi/PANi electrode surface in pyrrole containing acetonitrile + tetrabutyl ammonium perchlorate (TBAP) solution medium. The TBAP was found to be suitable as electrolyte in acetonitrile medium for homogeneous polypyrrole synthesis. As a result, the homogeneous and adherent PANi and top PPy films were synthesized on CS/ZnNi electrode surface. The corrosion performance of the bare CS electrode was compared with CS/ZnNi, CS/ZnNi/PANi and CS/ZnNi/PANi/PPy electrodes. The single ZnNi alloy coating and bilayer ZnNi/PANi coating did not show any significant barrier behavior on the CS electrode surface. On the other hand, the PPy coating as the top layer exhibited a significant physical barrier behavior on the CS/ZnNi/PANi electrode at longer exposure time.

Author Contributions:

IF performed the synthesis and corrosion measurements of the alloy and polymer coatings. ATO and IF evaluated the corrosion performance of the alloy and polymer films. IF wrote the manuscript.

ATO controlled the manuscript. All authors read and approved the final manuscript.

Conflict of interest disclosure:

The authors state that there are no conflicts of interest to disclose.

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