

Comparative analysis of corrosion resistance of Zinc and Zn-Al-Mg coatings on carbon steel

Análise comparativa da resistência à corrosão de revestimentos de Zinco e Zn-Al-Mg em aço carbono

Análisis comparativo de la resistencia a la corrosión de recubrimientos de zinc y Zn-Al-Mg sobre acero al carbono

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Abstract

One of the main ways to protect steel against corrosion is by the galvanizing process. This process has been continuously developed and its first optimization was performed by the addition of Aluminum in the coating. As a result, Zn-5wt.%Al and 55wt.%Al-Zn coatings were developed. Recently, the search for increased corrosion resistance led to the development of zinc coating with the addition of magnesium and aluminum. In this work, a comparative study of the corrosion resistance of zinc coated steel and Zn-2wt.%Al-1wt.%Mg alloy coated steel was performed. Samples were exposed to immersion corrosion tests in 0.1 M NaCl electrolyte and were analyzed by using SEM, XDR and EIS. On zinc-coated steel, the steel substrate was attacked after 48 days of immersion, while on Zn-2wt.%Al-1wt.%Mg coated steel, the steel substrate showed corrosive process after 90-day of immersion. The corrosion product formed from Zn-2wt.%Al-1wt.%Mg coated steel is the main cause of its better corrosion resistance compared to zinc coated steel.

Keywords: Coated Steel; Corrosion; Magnesium; Microstructure.

Resumo

Uma das principais formas de proteger o aço contra a corrosão é pelo processo de galvanização. Este processo tem sido continuamente desenvolvido e sua primeira otimização foi realizada com a adição de Alumínio no revestimento. Como resultado, foram desenvolvidos revestimentos de Zn-5% em peso de Al e 55% em peso de Al-Zn. Recentemente, a busca por maior resistência à corrosão levou ao desenvolvimento do revestimento de zinco com adição de magnésio e alumínio. Neste trabalho, foi realizado um estudo comparativo da resistência à corrosão do aço revestido com zinco e do aço revestido com liga de Zn-2% Al-1% Mg. As amostras foram expostas a testes de corrosão por imersão em eletrólito NaCl 0,1 M e analisadas por meio de MEV, XDR e EIS. No aço revestido com zinco, o substrato de aço foi atacado após 48 dias de imersão, enquanto no aço revestido com Zn-2% Al-1% Mg revestido em peso, o substrato de aço apresentou processo corrosivo após 90 dias de imersão. O produto de corrosão formado a partir de aço revestido de Zn-2% Al-1% em peso de Mg é a principal causa de sua melhor resistência à corrosão em comparação ao aço revestido de zinco.

Palavras-chave: Aço revestido; Corrosão; Magnésio; Microestrutura.

Resumen

Una de las principales formas de proteger el acero contra la corrosión es mediante el proceso de galvanizado. Este proceso se ha desarrollado continuamente y su primera optimización se realizó mediante la adición de Aluminio en el

recubrimiento. Como resultado, se desarrollaron revestimientos de Zn-5% en peso de Al y 55% en peso de Al-Zn. Recientemente, la búsqueda de una mayor resistencia a la corrosión condujo al desarrollo de un revestimiento de zinc con la adición de magnesio y aluminio. En este trabajo, se realizó un estudio comparativo de la resistencia a la corrosión del acero recubierto de zinc y el acero recubierto de aleación de Zn-2% en peso Al-1% en peso Mg. Las muestras se expusieron a pruebas de corrosión por inmersión en electrolito de NaCl 0,1 M y se analizaron utilizando SEM, XDR y EIS. Sobre acero revestido de zinc, el sustrato de acero fue atacado después de 48 días de inmersión, mientras que sobre acero revestido con Zn-2% en peso de Al-1% en peso de Mg, el sustrato de acero mostró un proceso corrosivo después de 90 días de inmersión. El producto de corrosión formado a partir de acero revestido con Zn-2% en peso Al-1% en peso Mg es la causa principal de su mejor resistencia a la corrosión en comparación con el acero revestido con zinc.

Palabras clave: Acero revestido; Corrosión; Magnesio; Microestructura.

1. Introduction

In order to improve corrosion resistance and reduce costs of galvanized steel sheet, great efforts have been invested into optimization of the zinc coating composition by alloying.

The zinc coating provides corrosion protection to steel by cathodic protection. In addition to the galvanic protection, the insoluble zinc corrosion products formed on the surface in atmospheric conditions assure a barrier effect (Yoo et al., 2014). Nevertheless, the composition of those corrosion products depends on the exposure conditions and this protection is not observed in all environments (Volovitch, 2009; Prosek, 2008; Hosking, 2007; Le Bozec, 2013; Salgueiro, 2015). In the last twenty years, a large effort has been made in order to improve the corrosion resistance of zinc coatings without increasing its thickness or resorting to additional corrosion protection such as protective layers. New metallic coatings alloying zinc with aluminum, magnesium or a combination of both elements have been the subject of numerous studies (Schuerz, 2009; Elvins, 2008; Prosek, 2010; Dutta, 2010; Li, 2012; Diler, 2012; Diler, 2014; Duchoslav, 2014; Persson, 2013; Salgueiro, 2015; Klemm, 2011; Thébault, 2015; Chen, 2010). Successful examples are coatings alloyed with aluminum, Zn-5%Al and Zn-55%Al. Nowadays, the main development activity is focused on Zn-Al-Mg coatings.

There are several studies about the effect of aluminum and magnesium alloys on corrosion resistance (Frankel, 1998; Kairy, 2016; Zander, 2007). Recently, researchers have evaluated the effect of adding magnesium and aluminum on the microstructure of the zinc coating and on the corrosive process (Krieg, 2014; Vimalanandan, 2014).

Prosek et al. (2016) in a recent study concluded that the mass losses of Zn-5Al and Zn-3Al-2Mg coatings varied up to 2-times as a function of their microstructure. In most cases, finer structures were more corrosion resistant, but the phase chemical composition apparently also played an important role. Prosek et al. (2016) suggested that zinc alloy with aluminum and magnesium reduced the corrosive process of zinc due to the stabilization of less soluble and more protective corrosion products. The formation of non-protective corrosion products has been reduced for these alloys. So, the better corrosion resistance of these alloys can be attributed to the availability of ions capable of forming protective corrosion products, which act as a physical barrier in the cathodic regions.

Dutta et al. (2010) analyzed the Morphology and properties of hot dip Zn-Mg and Zn-Mg-Al alloy coatings on steel sheet. They verified binary and ternary eutectic phase mixtures in the coatings formed in Zn-Mg and Zn-Mg-Al baths, respectively. The MgZn₂ formation was observed on the top surface and through the cross-section of the coating carried out in higher Mg bath. Steels coated in Zn-0.5%Mg-0.25%Al showed better corrosion resistance than steels coated in pure zinc and Zn-Mg.

Other authors have also demonstrated a corrosion resistance improvement of coatings based on zinc alloyed with magnesium by comparison to zinc coatings in atmospheric conditions.

In atmospheric conditions in the presence of chlorides, Hosking et al. (2007) attributed to Mg the ability to facilitate the formation of the protective simonkolleite layer (Zn₅(OH)₈Cl₂·H₂O). For their part, Volovitch et al. (2009) proved that

magnesium is also able to avoid the transformation of the protective simonkolleite layer into a mixed layer of smithsonite (ZnCO_3), hydrozincite ($\text{Zn}_5(\text{CO}_3)_2(\text{OH})_6$) and zincite (ZnO). Based on these studies, in atmospheric conditions and in presence of chlorides the corrosion reactions of Zn-Mg alloys involve the formation of simonkolleite which preserves the surface by opposition with other corrosion products of zinc. On the other hand, simonkolleite was not detected in corrosion products when the alloy is exposed to climatic conditions with and without contamination by chloride ions (Prosek, 2008; Diler, 2014), or during the early stages in atmospheric corrosion (Thébault, 2015; Chen, 2010).

Hosking et al. (2007) showed in his studies a 3-fold increase in time to red rust appearance of zinc–magnesium coated steel in a cyclic corrosion test compared to conventional zinc coatings. They attributed the prolonged life time of ZnMg coatings to the ability of magnesium corrosion products to neutralize hydroxide ions at cathodes creating conditions for insoluble simonkolleite to extend over the corroding surface, retarding thereby the overall corrosion reaction.

Yao et al. (2016) investigated the effect of Mg content on microstructure and corrosion of Zn-Al-Mg coatings. They attribute that the good corrosion resistance of ZAM coating comes from the flocculent type simonkolleite. The structure of simonkolleite prolongs the micro-path and impedes the movement of O_2 and H_2O , ultimately retarding the overall corrosion process of ZAM coating.

Rodriguez et al. (2016) studied the effect of the electrolyte pH on the corrosion mechanisms of Zn-Mg coated steel. The authors suggest that the initial value of the pH is a critical parameter in the corrosion mechanism. If the pH is higher than 11 at the beginning of immersion, the surface is passivated and stable with time. The passive layer is composed of hydrozincite $\text{Zn}_5(\text{CO}_3)_2(\text{OH})_6$ and magnesium carbonates (MgCO_3 and $\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2$). The formation of zincite is inhibited. The presence of chloride ions does not hinder the formation of a protective layer.

In other study, Schurz et al. (2010) found that Zinc–aluminum–magnesium coated steel offers a decisively enhanced corrosion resistance in salt spray test compared to conventional zinc coated steel. Even after short exposure times the entire metallic ZM coating is transformed into a stable, adherent aluminum-rich protecting layer identified as zinc aluminum carbonate hydroxide, $\text{Zn}_6\text{Al}_2(\text{CO}_3)(\text{OH})_{16}\cdot 4\text{H}_2\text{O}$. This long-lasting layer protects the steel substrate against corrosive attacks and is the main reason for the enhanced corrosion resistance of the ZM coating system.

The present work aims to study the effect of Mg and Al additions on the morphology and corrosion resistance of the zinc coatings on carbon steel substrate.

2. Methodology

2.1 Materials

The material for this study was supplied by CSN. Two coatings for steel sheet were investigated: the Zn coating consisting of Zn + 0.2 wt.% Al and Zn-Al-Mg coating consisting of Zn + 2wt.% Al + 1wt.% Mg. Both materials were produced in a continuous hot-dip galvanizing line. The average coating weight per side was about 135 g/m^2 for conventional Zn coating and Zn-Al-Mg coating, which corresponds to a coating thickness of $20 \mu\text{m}$. The steel substrate for both coating systems was a low carbon steel.

2.2 Electrochemical measurements

Zn coating and Zn-Al-Mg coating were studied by electrochemical impedance spectroscopy in order to evaluate the corrosion resistance of the coating as a function of immersion time in 0.1 M NaCl electrolyte at the natural pH. The stability of the open circuit potential (OCP) was previously checked for 20 minutes of immersion. EIS measurements were performed in a frequency range from 100 kHz to 10 mHz. All the spectra were recorded at OCP, applying 10 mV sinusoidal perturbation. A

conventional three-electrode electrochemical cell was used, composed of an Ag/AgCl/KCl saturated reference electrode, a platinum counter-electrode and a working electrode. The sample area in contact with the electrolyte was 0.78 cm² and the electrolyte volume was 70 ml. The cell was placed in a Faraday cage in order to avoid external interferences.

2.3 SEM and XRD

The morphology and composition of different phases were evaluated by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray mapping analysis, both on the top surface and across the cross-section of the coating. Samples of dimension 2 cm × 2 cm were cut out for X-ray diffraction (XRD) study to detect different phases present in the coating.

After 41, 56 and 90 days of immersion in 0.1 M NaCl electrolyte at the natural pH, Zn-Al-Mg coating were cleaned with demineralized water and dried with a dehydrated compressed air system. The metallic surface covered by corrosion products was characterized by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD).

3. Results and Discussion

The coating compositions were measured by inductively coupled plasma optical emission spectrometry (ICP-OES) and coating thickness were measured by SEM micrograph. The Table 1 shows the results of properties of the tested coatings.

Table 1. Properties of the tested coatings: Thickness and composition.

Materials	Thickness (μm)	Al(wt%)	Mg(wt%)	Zn(wt%)
Zn coating	20.3 ± 0.3	0.2	---	99.8
Zn-Al-Mg coating	21.1 ± 0.2	2.1	1.2	96.7

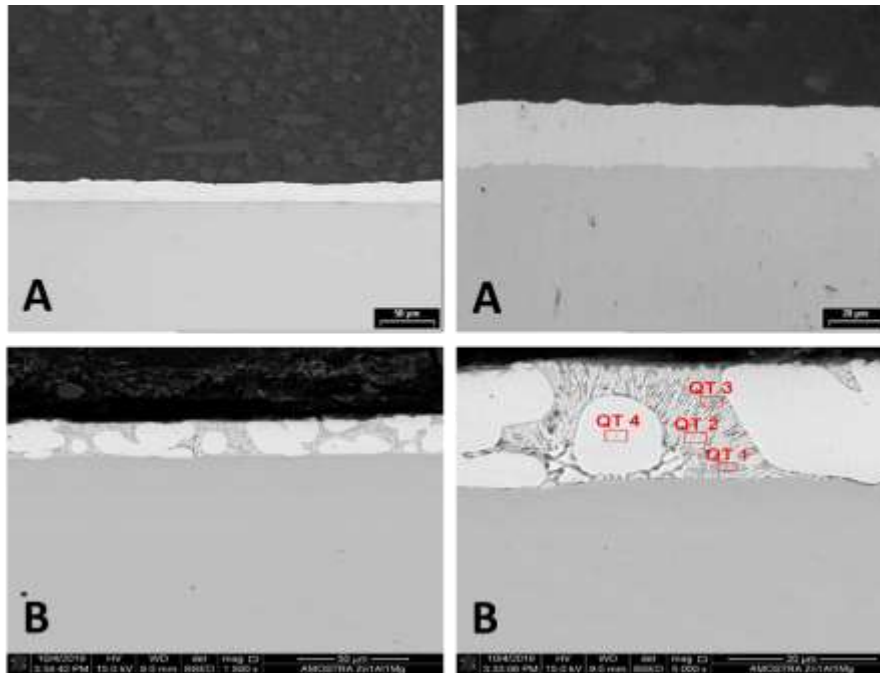
Source: Authors (2020).

3.1 Cross section Microstructure before immersion

Figure 1 shows the cross-sectional images of the Zn coating and the Zn-Al-Mg coating prior to the 0.1M NaCl solution immersion test. Table 2 presents the results of the quantitative analysis performed by EDS in the regions indicated in figure 1.

Zn coating has a homogeneous coating consisting of pure zinc and the Zn-Al-Mg coating has a coating formed by of zinc dendrite (phase η) – QT4, ternary eutectic consisting of Mg₂Zn₁₁, zinc and aluminum – QT2 and QT3 and small zinc aluminum particles – β phase (Prosek et al., 2016).

Figure 1. Cross-sectional SEM microstructures and EDS analyses for the (A) Zn coating and (B) Zn-Al-Mg coating.



Source: Authors (2020).

Table 2 shows the composition of the regions marked in Figure 1. The results obtained are like the work carried out by Prosek et al. (2016)

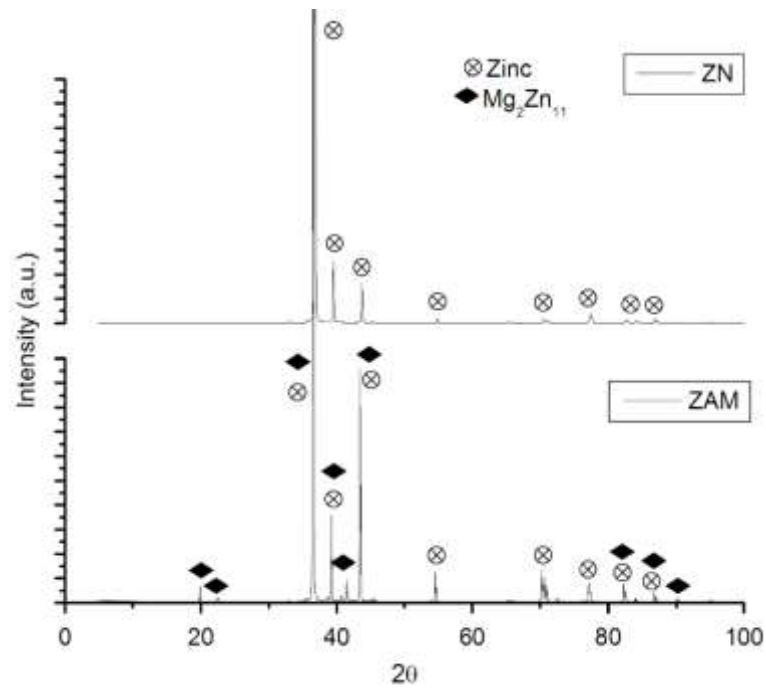
Table 2. EDS results of Zn-Al-Mg coating in the Figure 1.

	Zn (wt %)	Al (wt%)	Mg (wt %)
β phase – QT1	80.27	19.14	0.59
Ternary eutectic - QT2	91.19	6.55	2.27
Ternary eutectic - QT3	92.54	6.74	0.72
η phase – QT4	98.84	0.92	0.24

Source: Authors (2020).

Figure 2 shows the X-ray diffraction (XRD) analyzes performed on the surfaces of Zn coating and Zn-Al-Mg coating. It can be observed that in the Zn coating only the zinc element was detected, while the Zn-Al-Mg coating, besides the Zn element, was also detected the Mg_2Zn_{11} compound.

Figure 2. XRD analyses on Zn coating (ZN) and Zn-Al-Mg coating (ZAM) samples before immersion.



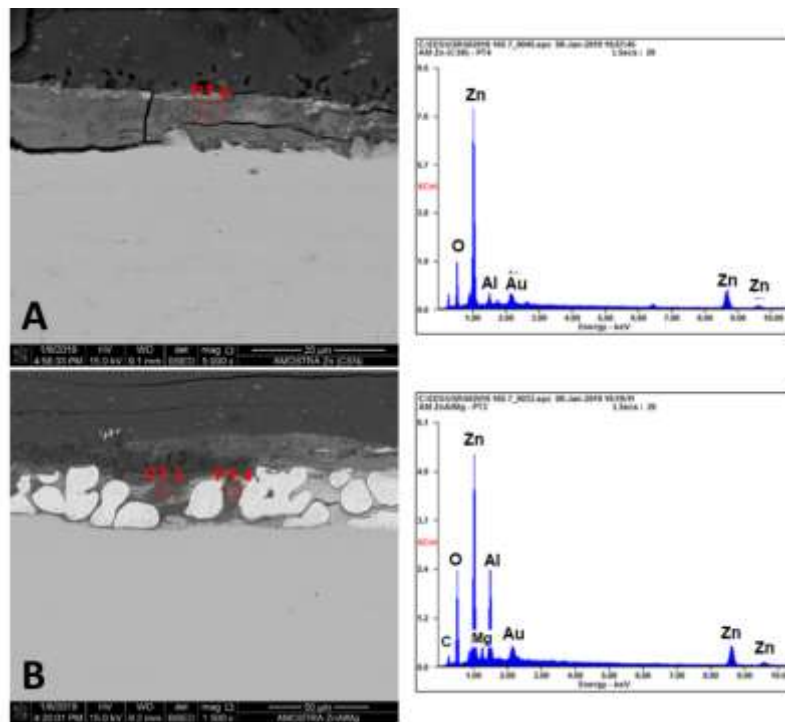
Source: Authors (2020).

3.2 Cross-section microstructure after immersion

Figure 3 shows the cross-sectional images of materials ZN and Zn-Al-Mg after corrosion of the base steel in the total immersion test in 0.1M NaCl solution. For ZN material, corrosion of the base steel occurred after 48 days of immersion, while for Zn-Al-Mg material after 90 days of immersion.

ZN coating showed uniform corrosive process throughout the zinc coating. For the Zn-Al-Mg coating, it is verified that the corrosive process in the coating was localized, occurring preferentially in the region composed by the ternary eutectic, with the preservation of the zinc dendritic regions.

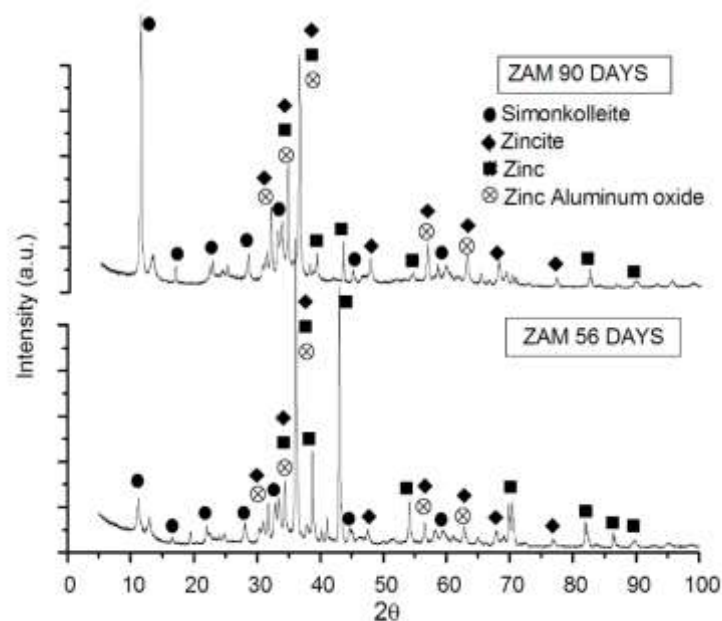
Figure 3. Cross-section microstructure and EDS after immersion. (A) Zn coating – 48 days immersion; (B) Zn-Al-Mg coating – 90 days immersion



Source: Authors (2020).

Figures 4 and 5 show the X-ray XRD diffraction analyzes on the surface of Zn coating and Zn-Al-Mg coating after immersion in 0.1M NaCl solution.

Figure 4. XRD analyses of corrosion products formed on Zn-Al-Mg coating (ZAM). 56 days and 90 days.

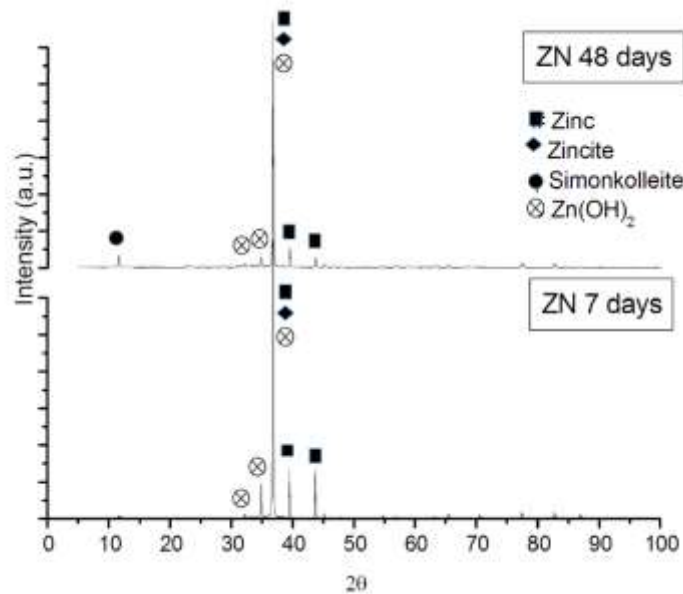


Source: Authors (2020).

According to Figure 4, the main corrosion products observed on the surface of the Zn-Al-Mg coating were simonkolleite ($Zn_5(OH)_8Cl_2H_2O$), Zinc oxide and Zinc-aluminum oxide. Simonkolleite is a compact compound that slows down the corrosive process of the coating.

XRD analysis of the ZN coating in Figure 5, shows that the main corrosion products observed on the material surface are Zinc oxide (zincite) and $Zn(OH)_2$. These corrosion products do not provide effective zinc coating barrier protection.

Figure 5. XRD analyses of corrosion products formed on ZN coating (ZN). 7 days and 48 days.

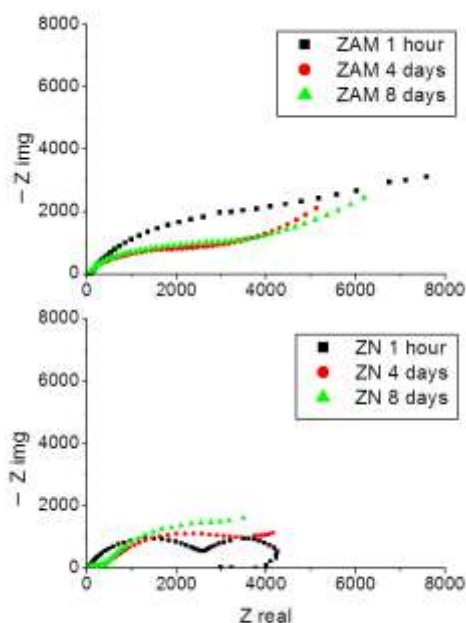


Source: Authors (2020).

3.3 EIS measurements

Figure 6 shows the Nyquist diagram of ZN coating and Zn-Al-Mg coating obtained after different immersion times (1 hour, 4 days and 8 days) in 0.1M NaCl solution.

Figure 6. Nyquist diagrams of EIS measurements recorded after 1h, 4 days and 8 days of immersion of Zn coating (ZN) and Zn-Al-Mg coating (ZAM) in 0.1M NaCl at natural pH (initial pH 5.6).



Source: Authors (2020).

For the ZN coating, it can be seen that the Nyquist diagram for 1 hour of immersion presented 2 capacitive arcs, the first arc obtained at high frequencies can be attributed to the formation of a corrosion product film on the surface of the ZN coating the while the second capacitive arc obtained at medium and low frequency can be attributed to charge transfer resistance of the electric double layer. For longer immersion times (4 and 8 days), only the formation of a capacitive arc can be attributed to the corrosion product film formed on the surface of the ZN coating, Yao et al. (2016).

For the Zn-Al-Mg coating, the Nyquist diagram presented only one capacitive arc at high and medium frequencies for all test times. This capacitive arc can also be attributed to the corrosion product film formed on the Zn-Al-Mg coating surface.

The values of the polarization resistance (corrosion product resistance) until the corrosion process of the base steel, are shown in Table 3, indicating that Zn-Al-Mg coating has better corrosion resistance than ZN coating. ZN coating showed red corrosion with 48 days of immersion in 0.1 M NaCl solution, while Zn-Al-Mg coating showed only 90 days.

Table 3. Results of fitting of EIS spectra recorded for ZN coating and Zn-Al-Mg coating immersed in 0.1M NaCl at the natural pH.

	$R_{pol. ZAM} (\Omega)$	$R_{pol. ZN} (\Omega)$
1 hour	6000	3000
4 days	2200	510
8 days	2500	290
19 days	3800	170
25 days	3200	90
48 days	2700	50
65 days	2200	-----
90 days	1800	-----

Source: Authors (2020).

4. Conclusion

It was concluded that Zn-Al-Mg coating showed higher corrosion resistance than Zn coating in 0.1M NaCl solution immersion test.

The formation of simonkolleite corrosion product on the surface of Zn-Al-Mg coating was responsible for its higher corrosion resistance compared to Zn coating.

There was no formation of simonkolleite in the zinc coating after immersion in 0.1M NaCl solution. Thus, we can attribute that the addition of aluminum and magnesium elements favored the formation of simonkolleite on the surface of the Zn-Al-Mg coating.

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