Pamukkale Üniversitesi Mühendislik Bilimleri Dergisi
Pamukkale University Journal of Engineering Sciences

Investigation of the corrosion properties of AA5754 aluminum alloy coated with graphene oxide by the electrophoretic deposition method

Elektroforetik biriktirme yöntemi ile grafen oksit kaplanmış AA5754 alüminyum alaşımının korozyon özelliklerinin incelenmesi

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Received/Geliş Tarihi: 21.10.2022 Revision/Düzeltme Tarihi: 23.02.2023 doi: 10.5505/pajes.2023.46027
Accepted/Kabul Tarihi: 24.02.2023 Research Article/Araştırma Makalesi

Abstract

This study aims to coat the aluminum alloy (AA5754) with graphene oxide by electrophoretic deposition (EPD) method, which is a cheap, eco-friendly, and straightforward process, and to determine the effect of the coating on the corrosion properties of the aluminum alloy. The graphene oxide coatings were characterized by X-ray diffraction (XRD), optical microscopy, and scanning electron microscopy (SEM). It was determined that the graphene oxide coatings grew layer by layer and their thickness increased with increasing time and applied potential. The effect of graphene oxide coating on the corrosion properties of AA5754 aluminum alloy was investigated by a potentiodynamic method in 3.5% NaCl environment. According to corrosion test results, it was determined that the corrosion potential of the coated AA5754 aluminum alloy changed positively, while the corrosion current and corrosion rate decreased. The highest corrosion potential was determined as -805 mV at 5 V-5 min and 7 V-3 min coating conditions. The lowest corrosion current density ($9.8.10^{-7}$ A·cm^{-2}), and corrosion rate (0.011 mm·y^{-1}) were obtained at 5 V-3 min. coating conditions.

Keywords: Graphene oxide, Aluminum, Electrophoretic deposition, Corrosion, Coating.

Öz

Bu çalışma, ucuz, çevre dostu ve basit bir işlem olan elektroforetik biriktirme (EPD) yöntemiyle, alüminyum alaşımını (AA5754) grafen oksit ile kaplamayı ve kaplamanın alüminyumun korozyon özelliği üzerindeki etkisini belirlemeyi amaçlamaktadır. Grafen oksit kaplamalar, X-ışını kırınımı (XRD), optik mikroskop ve taramalı elektron mikroskobu (SEM) kullanılarak karakterize edilmiştir. Grafen oksit kaplamaların katman katman büyüdüğü ve artan süre ve uygulanan potansiyel ile kalınlıklarının arttığı belirlenmiştir. Grafen oksit kaplamanın AA5754 alüminyum alaşımının korozyon özelliği üzerindeki etkisi, ağırlıkça %3.5 NaCl ortamında potansiyodinamik yöntemle araştırılmıştır. Korozyon test sonuçlarına göre, kaplanmış AA5754 alüminyum alaşımının korozyon potansiyelinin olumlu yönde değiştiği, korozyon akımının ve korozyon hızının ise azaldığı tespit edilmiştir. En yüksek korozyon potansiyeli 5 V-5 dk ve 7 V-3 dk. kaplama koşullarında -805 mV olarak belirlenmiştir. En düşük korozyon akımı yoğunluğu ($9.8.10^{-7}$ A·cm^{-2}) ve korozyon hızı (0.011 mm·y^{-1}) 5 V-3 dk. kaplama koşullarında elde edilmiştir.

Anahtar kelimeler: Grafen oksit, Alüminyum, Elektroforetik biriktirme, Korozyon, Kaplama.

1 Introduction

Aluminum alloys are used in many areas such as construction, automotive, aircraft, aerospace and defense industries due to their advantages such as lightness, high specific strength, and good ductility [1]-[7]. Some alloying elements used to increase the mechanical properties of aluminum can reduce corrosion resistance. Due to the low corrosion resistance, both the surface properties and mechanical properties of aluminum alloys deteriorate, and as a result, the service life of the material is shortened. Different coating processes, ceramic powder doping and surface treatments are applied by using various materials to improve the corrosion properties of aluminum alloys [8]-[10].

Graphene and graphene oxide (GO) are promising coating materials for corrosion applications, allowing coating from single-atom thickness to micron thickness. Graphene oxide coatings can provide high corrosion resistance even in very thin coating thicknesses by covering the material surface uninterruptedly [11]-[17]. This coating can be carried out by many different methods, including mainly chemical vapor deposition, spin coating, dip coating and electrophoretic deposition (EPD) [18],[19]. In recent years, the EPD method stands out among other methods due to its low cost, eco-friendly process, easy-to-control coating parameters, homogeneous coating and short coating times [11]-[16],[20], [21]. In the EPD method, the coating occurs in two stages: the suspended charged particles are transported to an oppositely charged electrode under the influence of the electric field and attached to the metal surface to form a compact film [22].

While many studies on graphene oxide coating by electrophoretic deposition on different materials, studies on graphene oxide coating of aluminum alloys are pretty limited. Graphene oxide coatings on various metals have been found to increase the corrosion resistance of the materials. Naghdi et al. [11] investigated the corrosion behavior of pure aluminum coated with graphene oxide with the EPD technique and stated that graphene oxide was very effective in protecting the aluminum surface in a chloride environment. Al-Sammarraie et al. [14], subjected the stainless steel, copper and aluminum surfaces to the corrosion test in a seawater environment by coating them with graphene oxide using the EPD method. In corrosion tests, they reported that coated samples showed better corrosion resistance than uncoated samples. Aliyu et al. [23] coated Fe-Mn on suspensions containing graphene oxide in different ratios with the EPD method and investigated the

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coating morphologies and corrosion behavior in 3.5% NaCl environment. It was found that the surface morphology and corrosion behavior of the coatings were sensitive to the graphene oxide content in the coating. When the graphene oxide concentration was increased up to a specific value, it was observed that the corrosion resistance increased, and the coating was compact, uniform and crack-free. After this value, it is stated that the protection efficiency decreases as a due to micropores and surface defects due to the inhomogeneous distribution of the stacked graphene oxide in the coating matrix.

The aim of this study; to improve the corrosion resistance of AA5754 aluminum alloy, it is to coat with graphene oxide, to characterize the coating and to determine the corrosion properties of the coating made with different parameters. EPD method, which is a cheap, environmentally friendly and simple method, was used in the graphene oxide coating process. Optimum coating parameters were determined by applying three different coating potentials (3 V, 5 V and 7 V) and three different coating times (1 min., 3 min. and 5 min.). The applied parameters were selected according to the literature and preliminary experiment results. Optical microscope, SEM with EDS analysis and XRD were used for coating characterization. The corrosion properties of AA5754 aluminum alloy coated with graphene oxide with different parameters were investigated by using an electrochemical potentiodynamic method, in 3.5% NaCl environment. The effects of different coating parameters on the corrosion rates of graphene oxide coated aluminum alloy were determined.

2 Experimental

2.1 Electrophoretic deposition process

AA5754 aluminum alloy was 2 mm thick plate and was cut in 10 x 100 mm size for use in coating and corrosion studies. In graphene oxide (GO) coating studies, 1 cm^2 surface area of AA5754 aluminum alloy was coated. The chemical composition of the AA5754 aluminum alloy used in the experiments is given in Table 1. Before coating, aluminum alloy samples were firstly grinded with silicon carbide sandpaper, cleaned with acetone and ethanol in an ultrasonic bath, finally washed with distilled water and made ready for coating. Coating studies were carried out in a suspension of graphene oxide with a C·O atomic ratio is ~1.5 and a concentration of 0.5 $mg \cdot ml^{-1}$ in distilled water. In the electrophoretic coating process, a two-electrode glass cell was used (Figure 1), AA5754 aluminum alloy as the anode and a copper plate as the cathode were placed at a distance of 10 mm between them. The GO deposition process on the aluminum alloy surface was carried out at room temperature by applying three different potentials (3, 5 and 7 V), and three different times (1, 3 and 5 minutes).

2.2 Characterization of graphene oxide coating

The microstructure and thickness of the graphene oxide coatings obtained with EPD were determined by Nikon ECLIPSE LV150N light microscope. The surface morphologies and content of the coatings were investigated using the JEOL JSM 6060 LV scanning electron microscope. Bruker D8 XRD device was used to determine the phase structures of graphene oxide coatings.

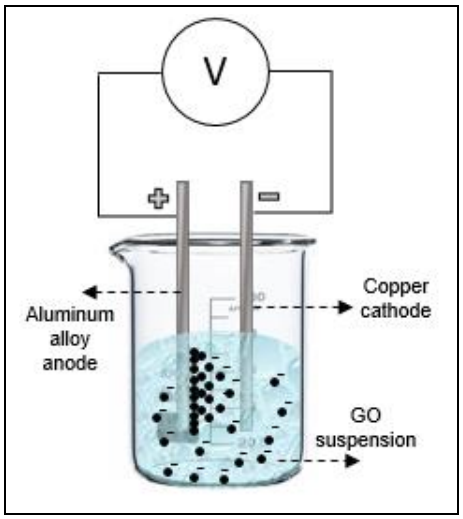

Figure 1. Schematic representation of the experimental cell.

2.3 Corrosion analyses

In the corrosion tests, uncoated and graphene oxide coated AA5754 aluminum alloy with a surface area of 1 cm^2 was used as the working electrode. Reference electrode was saturated calomel electrode and counter electrode was platinum. Electrochemical corrosion measurements were carried out in 3.5% NaCl solution with the help of Ivium Technologies de Regent potentiostat-galvanostat device. The potentiodynamic method was used in corrosion studies. In the first stage of corrosion studies, polarization curves were obtained by scanning from cathodic to anodic direction at a scanning rate of 50 $mV \cdot s^{-1}$ and a potential range of -1.8 V and -0.5 V. In the second stage, the corrosion potential (E_{corr}), corrosion current density (I_{corr}) and corrosion rate (CR) of the material were determined by obtaining the Tafel polarization curves at a scanning rate of 2 $mV \cdot s^{-1}$ in the same potential value range.

3 Results and discussion

3.1 Graphene oxide coating of AA5754 aluminum alloy

In the electrophoretic coating process, as a result of the potential applied in the coating cell, the negatively (–) charged graphene oxide particles suspended in distilled water move with the help of the electric field force towards the positively (+) charged AA5754 aluminum alloy substrate and forms a film on the aluminum alloy surface. Conductivity, zeta potential, concentration and electrochemical parameters are effective in the electrophoretic deposition of graphene-based materials. Graphene oxide can be deposited in different forms such as continuous film, fiber or irregular and porous layers. These forms are dependent on EPD process conditions, and mostly, a layer-by-layer structure is observed [16]. Graphene oxide coating formation was investigated at 3, 5, 7 V cell potentials and 1, 3, 5 min coating times conditions and characterized in detail. In order to determine the stability of the suspension, zeta potential was measured before the coating process. The value of the zeta potential was obtained as -43.3 mV. This value can be considered “fairly good stability” [24].

Table 1. Chemical composition of AA5754 aluminum alloy (wt.%).

Fe	Si	Cu	Mn	Mg	Zn	Cr	Ti	Others	Al
0.4	0.4	0.1	0.5	2.6-3.6	0.2	0.3	0.15	0.15	Balance

Firstly, the phase structure of the formed coating was investigated. For this, graphene oxide layer formed at 5 V cell potential and 5 min. coating time conditions was examined by XRD analysis Figure 2(a). In the XRD pattern, the characteristic peak of the graphene oxide coating occurred at approximately 10°. Obtaining aluminum peaks as well as graphene oxide peaks is due to the thin structure of the coating.

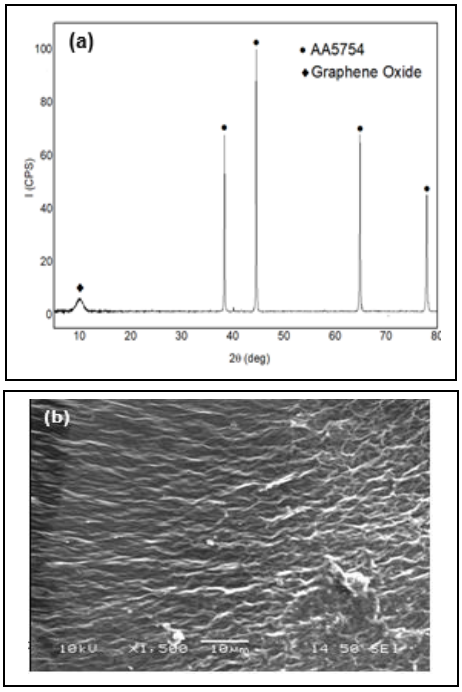

Figure 2(a): XRD diffraction pattern. (b): SEM image of AA5754 coated with GO (5 V-5 min).

The coating surfaces of the AA5754 aluminum alloy, which was coated with graphene oxide by applying different potentials and different times, were examined with a SEM-EDS. SEM images of some of the AA5754 aluminum alloys surface coated with graphene oxide using 5 V-5 min and 3 V-1 min parameters are given in Figure 2(b) and Figure 3(a), respectively.

In the microstructure examinations made by SEM, it was shown that the surface of the aluminum surface was completely covered with graphene oxide at high potentials and times Figure 2(b). However, it has been determined that there are coating defects in the form of cracks on the surface and areas where the aluminum substrate is not completely closed during low potential and especially short coating times. The SEM image of the 3 V-1 min sample with such areas and the point EDS analysis applied to the coated and uncoated regions are given in Figure 3(b) (zone 1) and Figure 3(c) (zone 2). C and O elements, which are graphene oxide components, are not found in the EDS analysis performed in the light-colored coating defect region (zone 2, 0% C and O), which is seen especially in graphene oxide coated samples at low times, however, Al element is present in very trace amounts (4.121 and 6.131%) in the completely covered areas of graphene oxide (zone 1 and 3). Similar surface morphologies were also obtained in studies by Al-Sammarraie et al. [14], Ma et al. [16] and Chavez-Valdez et al. [19].

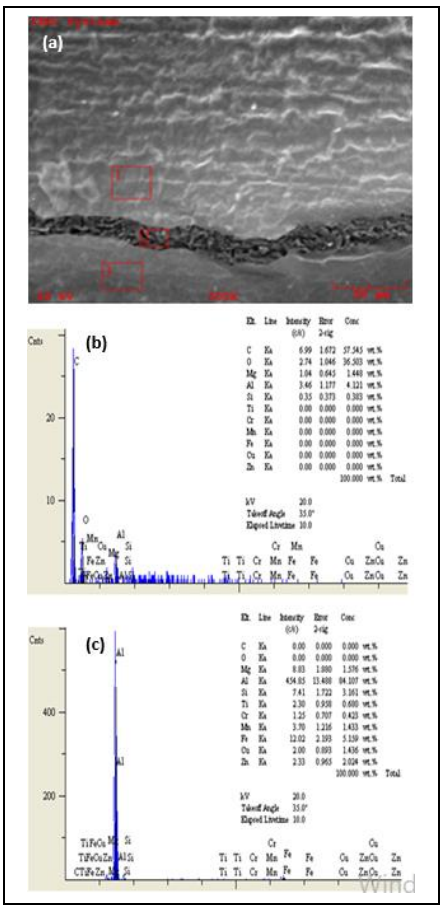

Figure 3(a): SEM image. (b): EDS analysis of surface coating zone 1. (c): Failure zone 2 (3 V-1 min sample).

In order to obtain information about the thickness of the graphene oxide coatings applied to the surface of the AA5754 Aluminum alloy and the uniformity of the coating, cross-sectional microstructure images of the samples were examined with a light microscope. In Figure 4, the microstructure images of AA5754 Aluminum alloy samples, which were coated for 3 minutes at different potential values, taken from cross-sections, are given. In these images, it is seen that the coating film is formed in a uniform thickness on the entire surface.

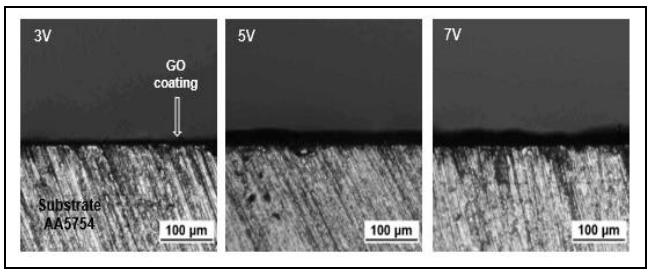

Figure 4. Cross-sectional images of AA5754 aluminum alloy coated for 3 min at 3, 5 and 7 V.

And cross-sectional images of coated samples were given in Figure 5, for 5 V at different times. It is seen that the coating thicknesses increase with increasing time.

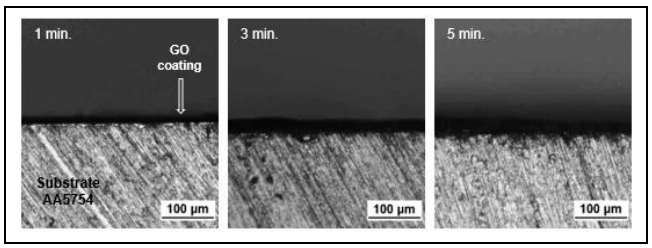

Figure 5. Cross-sectional images of AA5754 aluminum alloy coated at 5 V for 1, 3 and 5 min.

The average coating thicknesses of the samples coated at different potentials and times were obtained from the cross-section microstructure images are given in Table 2 and Figure 6. According to Table 2, there was an increase in coating thickness due to the increasing potential. When the potential was increased from 3 V to 5 V, there was no significant increase in coating thickness. However, as can be clearly seen in Figure 6, when the potential was increased to 7 V, the coating thickness increased more significantly. While the average coating thickness was around 20 µm in the 3 and 5 V potential applications in 1 min coating time, the average coating thickness was approximately 35 µm when the 7 V potential was applied for the same time. When coating thicknesses are evaluated in terms of coating times, increasing the coating time from 1 min to 3 min for 3 V and 5 V did not make a significant difference in coating thickness (Figure 6). In addition, it was determined that the coating thickness increased at a higher rate when the coating time was increased to 5 minutes. On the other hand, as the time increased, the coating thickness increased more regularly in the coatings performed at 7 V. However, in the coatings obtained by applying 7 V potential, it was determined that the uniform structure of the coating was deteriorated and it was out of the desired standard deviation (Figure 6).

Table 2. Average coating thicknesses (µm) obtained at different potentials and times.

	Coating time (min)		
Potential (V)	1	3	5
3	19.03	19.9	29.56
5	20.34	20.31	31.36
7	34.79	38.54	40.01

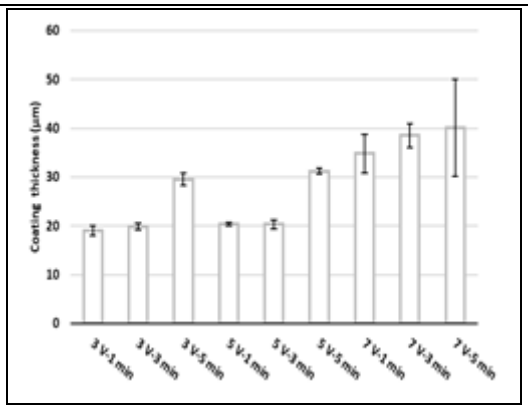

Figure 6. Coating thicknesses of the samples

There are few articles examining the effects of applied potential and time on coating thickness in the electrophoretic process. The results of these articles are similar to our study. Diba et al. [25] investigated the electrophoretic deposition kinetics of graphene oxide and reported that the coating thickness increased with the increase of the deposition time. Hwang et al. [26] and Reza et al. [27] reported that the coating thickness increased due to the increase in the deposition efficiency with the increase in the deposition time and deposition potential in their study where they coated graphene oxide on the copper surface by electrophoretic deposition method. Ho et al. [28], Park et al. [29] and Mahmoudi et al. [30], in their study where they coated graphene oxide on steel by electrophoretic method, determined that when the deposition time and deposition potential increased, the deposition rate increased and as a result, the coating thickness increased. Dinesh et al. [31] applied graphene oxide coating to NiTi alloy by electrophoretic method, and the coating thickness increased when the coating time was increased. As reported in these studies, with the increase of applied potential and/or time, the deposition mass and deposition rate of graphene oxide on the metal surface increases, and as a result, the coating thickness increases.

3.2 Corrosion behavior of graphene oxide coated AA5754 aluminum alloy

Graphene oxide coatings, which do not react with acid and corrosive brines, can improve the corrosion properties of metallic materials. For this reason, the effect of coatings formed at different potentials and times on the corrosion behavior of the aluminum alloy was investigated in a corrosive 3.5% NaCl environment. The polarization curves of uncoated and graphene oxide coated (at 5 V potentials and different times) AA5754 aluminum samples are given in Figure 7 and Figure 8, respectively. When the polarization curves are examined, it is seen that the current density is at very low levels approximately -1800 mV cathodic potential to -650 mV potential value in all samples. The region where the slope in the graph flattens is the passivation region for coated and uncoated AA5754 aluminum alloy. Resistance to corrosion is provided by the passive oxide film formed on the alloy surface in this region.

Although the aluminum surface is exposed in uncoated areas on the coating surface, the same corrosion resistance occurs due to passivation in this potential range. In all uncoated and graphene oxide coated samples, passivation ends and a sudden increase in current density values begins when the potential value of –650 mV is passed to the higher potential region, that is the anodic region.

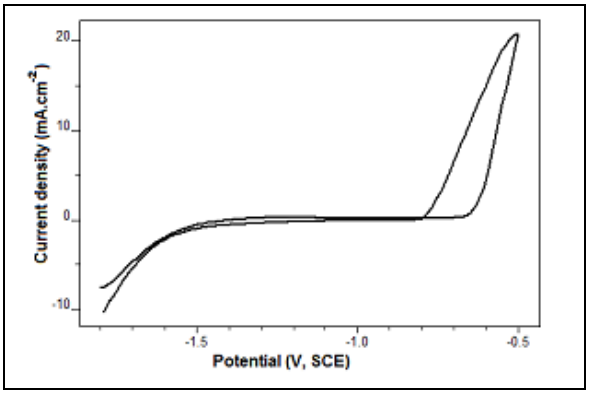

Figure 7. Polarization curve of uncoated AA5754 aluminum alloy.

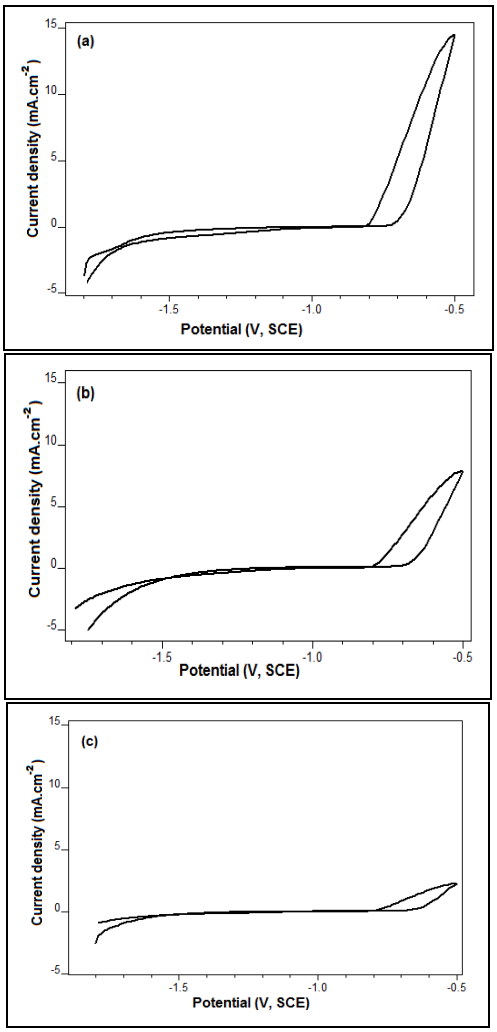

Figure 8. Polarization curves of GO coated AA5754 at 5 V potential for (a): 1 min. (b): 3 min. (c): 5 min.

This region is the transpassive region and the potential value in this region has been increased up to −500 mV. At this potential value, in the uncoated AA5754 aluminum alloy, the current density goes up to 20 mA·cm^{-2}, while at different potentials it is 13-15 mA·cm^{-2} in samples coated with graphene oxide for 1 min, 7-10 mA·cm^{-2} in samples coated for 3 min, and 2-4 mA·cm^{-2} in samples coated for 5 min.

In other words, lower current density levels were obtained in the graphene oxide coated samples. This indicates that the graphene oxide coatings applied to the AA5754 aluminum alloy surface provide a resistance against corrosion.

Tafel polarization curves of AA5754 aluminum alloy materials that are not coated and graphene oxide coated in different parameters are given in Figure 9, and some numerical corrosion data obtained from these curves are given in Table 3. When the effects of the graphene oxide coating made at different potentials on the corrosion properties were examined, the corrosion potential (E_{corr}) of the uncoated AA5754 aluminum alloy increased positively after the coating, according to Figures 9(a), Figure 9(b), Figure 9(c) and Table 3. The positive increase in E_{corr} creates a positive effect in terms of corrosion resistance. Corrosion current density (I_{corr}) values decreased in graphene oxide coated aluminum alloys.

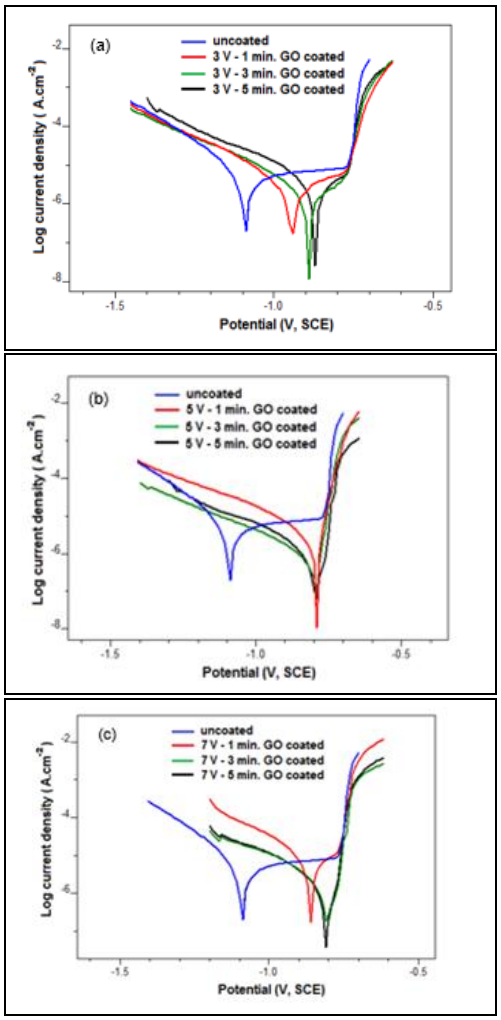

Figure 9. Tafel polarization curves of uncoated and GO coated AA5754 at different potentials and times for (a): 3 V. (b): 5 V. (c): 7 V.

A decrease in I_{corr} indicates an increased resistance of the material to corrosion. In the corrosion rate calculations obtained using I_{corr} values, the corrosion rate value of the uncoated aluminum alloy was 0.054 mm·y^{-1}, while the corrosion rate value decreased to 0.011 mm·y^{-1} and 0.014 mm·y^{-1}, especially in the coatings performed at 5 V, 3 and 5 minutes.

Table 3. Corrosion values of AA5754 uncoated and GO coated.

Potential (V)	Time (min)	E_{corr} (mV)	I_{corr} (A·cm^{-2})	CR (mm·y^{-1})
Uncoated	Uncoated	−1087	4.9.10^{-6}	0.054
3	1	−928	2.4.10^{-6}	0.026
3	3	−894	2.1.10^{-6}	0.023
3	5	−871	3.7.10^{-6}	0.040
5	1	−793	3.2.10^{-6}	0.035
5	3	−817	9.8.10^{-7}	0.011
5	5	−805	1.2.10^{-6}	0.014
7	1	−856	4.1.10^{-6}	0.045
7	3	−805	1.6.10^{-6}	0.017
7	5	−808	1.3.10^{-6}	0.015

The decreasing in corrosion rate in aluminum alloys coated with graphene oxide indicates that the graphene oxide coating acts as a barrier between aluminum and the saltwater environment. Naghdi et al. [11] reported a reduction in

corrosion rate as a result of the graphene oxide coating, and that the graphene oxide coating inhibits ion and electron transfer between aluminum and the brine electrolyte. It can be said that the same effect was observed in this study as well. As a result, the graphene oxide film deposited on the surface is a good barrier against the diffusion of oxygen and water to the metal surface.

The barrier property of the graphene oxide coated aluminum alloy surface against the environment is improving. Because the graphene oxide coating has many distinctive features such as gas impermeability, chemical (acid/base/salt environment) resistance, antibacterial potential, thermal stability, environmental friendliness, and most importantly, high specific surface area. Graphene oxide coatings can simultaneously exhibit an outstanding barrier property for reactive gases, liquids, salts and acids [32-34]. In addition, the defect-free graphene monolayer shows high resistance to aggressive acids such as hydrofluoric acid and aggressive anions such as chloride. Graphene oxide with reactive oxygen functional groups can behave as negatively charged species when exposed to an electrolyte such as NaCl environment. Therefore, in environments such as the brine solution used in this study, the electrostatic repulsion between negative charges in graphene oxide and electrolyte anions can block the access of anions responsible for corrosion formation to a metal surface. This inhibition also provides corrosion resistance. The graphene oxide coating also helps to protect the passive Al_2O_3 layer under high potential [35],[36].

4 Conclusion

In this study, AA5754 aluminum alloy was coated with graphene oxide by EPD method by applying different potentials (3, 5, 7 V) and different times (1, 3, 5 min.). The characterization of the graphene oxide coating was made and corrosion behavior of this graphene oxide coated alloy was investigated by electrochemical methods, and the following results were obtained;

a) Continuous and constant thickness graphene oxide coating on aluminum alloy under mild conditions was obtained by EPD method,

b) At low potentials and times, such as 3 V-1 min, it was discovered that there were uncoated areas on the aluminum surface. By increasing the potential to 5 V and the time to 3 minutes, the entire aluminum surface was covered,

c) Graphene oxide coating thickness increased with increasing time and potentials. The lowest coating thickness was obtained, 19.03 µm at 3 V cell potential in 1 min time conditions, and the highest coating thickness was obtained, 40.01 µm at 7 V potential, 5 min time conditions,

d) In corrosion tests performed in a 3.5% NaCl environment, the corrosion potential of graphene oxide coated AA5754 aluminum alloy changed positively, and the corrosion current density and corrosion rate decreased,

e) The highest corrosion potential was-805 mV at 5 V-5 min and 7 V-3 min coating conditions, and the lowest corrosion current density and corrosion rate were $9.8.10^{-7}$ A·cm^{-2} and 0.011 mm·y^{-1} at 5 V-3 min coating conditions, respectively.

5 Acknowledgment

This study was supported by Hitit University Scientific Research Projects Unit. Project No: MUH19001.20.003.

6 Author contribution statements

Duygu CANDEMİR provided the literature search, procurement of materials, performance of experiments and analyses and contributed during the writing phase of paper. Kubilay KARACİF acted as the coordinator of the study project, evaluated the results of the experiments and controlled the writing. Levent KARTAL assisted in the construction of the experiments and contributed to the writing.

7 Ethics committee approval and conflict of interest statement

There is no need to obtain permission from the ethics committee for the article prepared.

There is no conflict of interest with any person / institution in the article prepared.

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