Full Paper

Poly(2-anisidine) Coating on Aluminum Alloy 5052: Electrosynthesis, Characterization and Its Corrosion Protection Properties in Marine Environment

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Abstract- The electrosynthesis of poly(2-anisidine) on aluminum alloy 5052 (AA5052) surface was carried out by a galvanostatic method. Various constant current densities of 5, 10, 15 and 20 mA cm^{-2} at deposition times of 900, 1800, 2700 and 3600 s were applied to the formation of the coatings. The results showed that the current density of 15 mA cm^{-2} at the deposition time of 2700 s for the polymerization stage is the best condition for the synthesis of more compact and strongly adherent coatings. The coatings were characterized by FT-IR, UV-Vis and scanning emission microscopy (SEM) techniques. The corrosion protection performance of the coatings was investigated in 3.5% NaCl solution as a corrosive environment by the potentiodynamic polarization technique and electrochemical impedance spectroscopy (EIS). The corrosion current decreases significantly from 5.338 µAcm^{-2} for uncoated electrode to 0.004 µAcm^{-2} for coated electrode (under optimal conditions). The results clearly ascertain that the poly(2-anisidine) has outstanding potential to protect the AA5052 alloy against corrosion in a chloride environment.

Keywords- Poly(2-anisidine), Coating, Aluminum alloy 5052, Corrosion, Electrosynthesis

1. INTRODUCTION

Aluminum is commercially important metal and its alloys are widely used in many industries such as reaction vessels, pipes, machinery and chemical batteries because of their

advantages. Aluminum is an active metal and its resistance to corrosion depends on the formation of the protective oxide film [1]. The breakdown of the passive oxide film on aluminum and its alloys by aggressive sufficiently positive anodic potentials is frequently responsible for the failure of aluminum and its alloys in aqueous halide solutions, as it usually results in severe pitting of the underlying metal. Many different corrosion mechanisms are suggested for aluminum alloys. The most common types are generally well understood. For each, the process is complex, incorporates many factors, and varies according to metal and specific operating conditions. Yet all still remain difficult to control, and represent a very serious threat to most industries.

There are several methods to protect the aluminum and its alloys from corrosion such as coatings (metallic, inorganic, conversion and organic coatings), control of the environment and corrosion inhibitors (organic and inorganic additives).Organic coating provides protection by the formation of a barrier action from the layer and surface condition of metal converted to a more stable state by coating with organic compounds. These coatings delay the generation of electromotive force, causing the corrosion of the substrate [2]. Among the conducting polymers, polyaniline and its derivatives are found to be one of the most promising materials because of ease of synthesis, high electrical conductivity, nontoxic property, good environmental and chemical stability, chemical redox reversibility, and low cost [3-5].

One of the main problems with the use of conducting polymers for metal corrosion inhibition is their water permeability, which can lead to corrosive species being transported to the metal surface. Therefore, incorporation of hydrophobic functional groups is expected to improve the polymer protection efficiency [6,7].

2-anisidine is a substituted derivative of aniline with a methoxy ($-OCH_3$) group at the ortho position, therefore, this study explores the possibility of using poly(2-anisidine) as an alternative to polyaniline for the corrosion protection of aluminum. The monomer of 2-anisidine is commercially available at low cost and it has a quite good solubility in water. The electrochemical synthesis of poly(2-anisidine) from an aqueous bath also may provide an alternative for reducing the use of hazardous chemicals as well as the cost of waste disposal. The conversion of the monomer to the polymer is a straight forward process.

Recently, many attempts have been carried out to protect the aluminum and its alloys by polyaniline and its derivatives base coatings. Researchers were chemically or electrochemically synthesized these coatings on various aluminum alloys such as aluminum alloy 1050 (AA1050) [8,9], AA2024 [10], AA3003 [11], AA3004 [12], AA3104 [13], AA3105 [14], AA5083 [15], AA6061 [16], AA7075 [17], etc. Due to different applications of various aluminum alloys, the studies on the specific aluminum alloys are important and the obtained results will be useful for industries.

To the best knowledge of authors, a few studies have been reported on the corrosion protection of aluminum alloy 5052 using the polyaniline and its derivatives coatings. The 5xxx series aluminum alloys are commonly used in marine applications where low density materials, good mechanical properties and better resistance to corrosion are desired [18-20]. Of the commercial alloys, the Al–Mg alloys (5xxx grades) display a fairly good resistance against both general and localized corrosion in seawater and aqueous chloride-containing solutions.

The main objective of the present study is to electrosynthesispoly(2-anisidine) on AA5052 electrode from an aqueous oxalic acid solution via the galvanostatic method with optimizing the electrodeposition conditions. In this paper, we report the electrosynthesis of the adherent poly(2-anisidine) coatings on AA5052 using the electrochemical galvanostatic method. The coatings were then characterized by FT-IR, UV-Vis and SEM techniques. The corrosion resistant properties of the coated samples with poly(2-anisidine) were then evaluated with polarization and EIS techniques in 3.5% NaCl solution.

2. EXPERIMENTAL SECTION

All chemicals were purchased from Merck. 2-anisidine was freshly distilled and stored in the dark. All solutions were freshly prepared from analytical grade chemical reagents using doubly distilled water. For each run, a freshly prepared solution was used, and all experiments were carried out at room temperature.

Electrochemical experiments and corrosion tests were carried out using a μ-AUTOLAB potentiostat/galvanostat model μш AUTO71174 connected to a Pentium IV personal computer through a USB electrochemical interface. A conventional three-electrode cell was used.

The working electrode employed was prepared from an AA5052 sheet with the chemical composition (wt.%) of: Si (0.25), Fe (0.40), Cu (0.10), Mn (0.10), Mg (2.40), Zn (0.10), Cr (0.25) and Al (96.40). The metal sheet was cut into rectangular samples of 1 cm^2 area, soldered with Al-wire for an electrical connection and mounted onto the epoxy resin to offer only one active flat surface exposed to the corrosive environment. Before each experiment, the working electrode was abraded with a sequence of emery papers of different grades (200-2500), degreased with acetone, and then immersed in 5% NaOH solution for 2 minutes for activating the electrode surface. After this stage, the electrode washed thoroughly with running water and dipped in a concentrated H_3PO_4 solution for 30 s. The electrode was then washed with distilled water, and the pre-treated electrode used for electropolymerization stage.

Electropolymerization was carried out by the galvanostatic method with 10 mL solution included of the 0.5 M oxalic acid and 0.2 M 2-anisidine. Electrosynthesis of poly(2-anisidine) coatings over the AA5052 surface were carried out by imposing a fixed current density for a

certain deposition time. In this regard, current densities of 5, 10, 15 and 20 mA cm^{-2} were applied for 900, 1800, 2700 and 3600 s. In all cases, the corresponding potential transients were recorded.

Pre-treated AA5052 alloy was used as the working electrode in the conventional three-electrode assembly, having a Pt sheet as the counter electrode and an Ag/AgCl (3 M KCl) electrode as the reference electrode.

The FT-IR spectra of poly(2-anisidine) electrosynthesized on AA5052 was obtained using a Shimadzu Varian 4300 spectrophotometer in KBr pellets. The obtained poly(2-anisidine) was dissolved in pure N-methylpyrrolidone (NMP) and UV–Vis spectra of this polymer solution was recorded on a Perkin Elmer Lambda2S UV–Vis spectrometer. The morphology of the poly(2-anisidine) coatings was analyzed using a SERON model AIS-2100 scanning electron microscope instrument operating at 10 kV. The samples were mounted on a double-sided adhesive carbon disc and sputter coated with a thin layer of gold to prevent sample charging problems.

The poly(2-anisidine)-coated AA5052 samples were evaluated for their corrosion resistance properties by Tafel polarization and electrochemical impedance spectroscopy (EIS) after 1 h immersion in corrosive environments at room temperature. The working electrode was first immersed in the test solution for 60 min to establish a steady state open circuit potential (OCP).

In the case of Tafel polarization, the working electrode potential was scanned on the potential range of ±200 mV versus OCP with scan rate of 0.5 mV s^{-1}. From the anodic and cathodic polarization curves, the Tafel regions were identified and extrapolated to the corrosion potential (E_{corr}) to obtain the corrosion current density (i_{corr}) using the NOVA 1.6 software. In the case of electrochemical impedance spectroscopy, a.c. signals of 10 mV amplitude and various frequencies from 100 kHz to 0.1 Hz at open circuit potentials were impressed to the coated AA5052 in the electrode surface (1 cm^2). A Pentium IV powered computer and NOVA 1.6 software was applied for analyzing impedance data.

3. RESULTS AND DISCUSSION

3.1. Electrochemical synthesis of poly(2-anisidine) at constant current density

Adherent, homogenous and green poly(2-anisidine) coatings were successfully electrosynthesized on AA5052 at constant current density, from 0.5 M oxalic acid solutions containing 2-anisidine (0.2 M). The galvanostatic curves obtained for the electrochemical synthesis of poly(2-anisidine) at different applied current densities are shown in Fig. 1. An initial rapid increase in potential is observed and then, after a short period of time (that can be considered as an induction time), the potential reaches a constant value. The induction time was found to be depending on the applied current density.
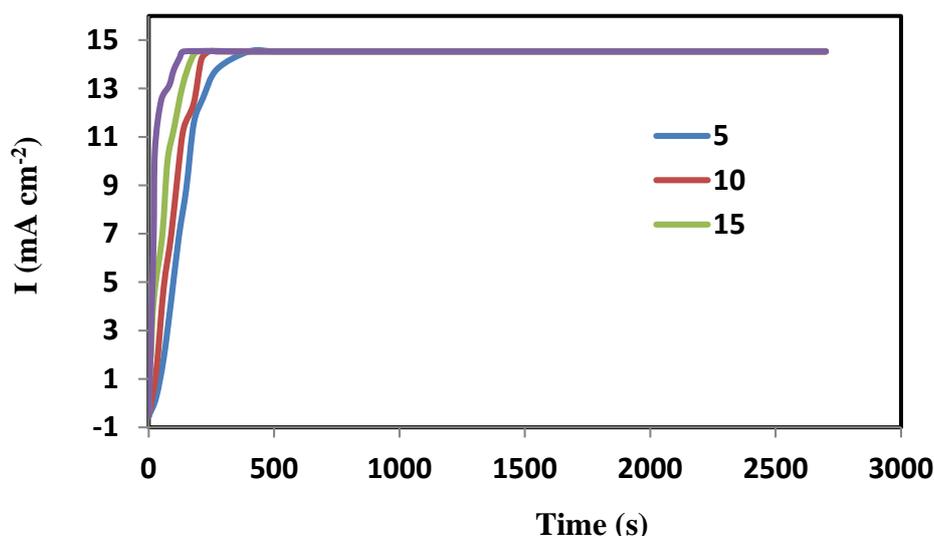
Fig. 1. Galvanostatic potential–time plots recorded for aluminum alloy 5052 in a 0.5 M oxalic acid +0.2 M 2-anisidine solution at different applied current densities

Smaller induction times are needed for higher applied current densities (Fig. 2). More Al^{3+} is produced per unit time at higher applied current densities with concomitant precipitation of more Al oxalate crystals on the substrate. Thus, the substrate could be covered by the passive Al oxalate layer in a shorter time. This passive layer inhibits the further dissolution of Al^{3+} without affecting the other electrochemical processes. In other words, the electrode surface behaves like an inert metal [21].
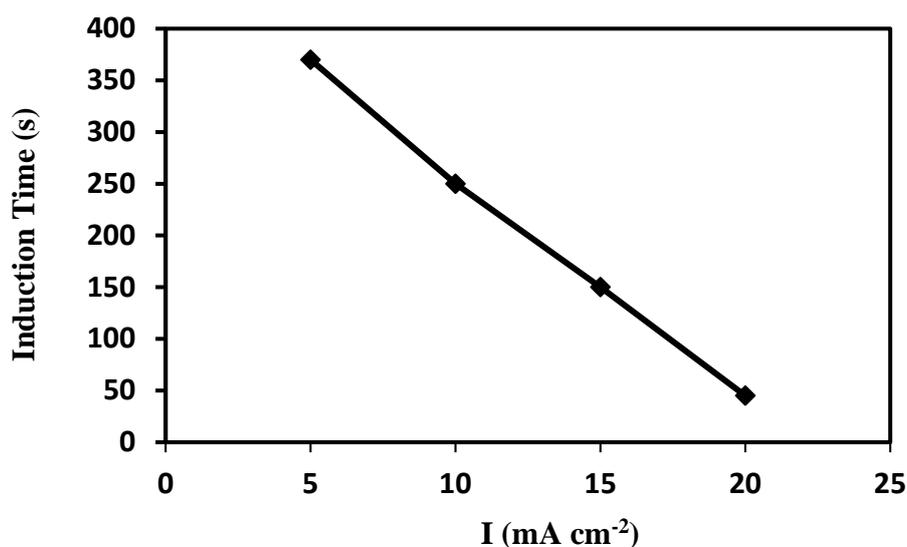

Fig. 2. Induction time as function of the electropolymerization current density

3.2. Spectroscopic characterization

Fig. 3 shows the UV–visible spectra in pure NMP solvent for poly(2-anisidine) coatings obtained under galvanostatic conditions on the surface of AA5052. Fig. 4 shows the FTIR spectrum for poly(2-anisidine) deposited on AA5052 electrode. As seen in Figs. 3 and 4, both spectra are compatible to literatures [22,23].The green coatings, characteristic of poly(2-anisidine) in the emeraldine oxidation state, were produced on the surface of AA5052 using galvanostatic technique.
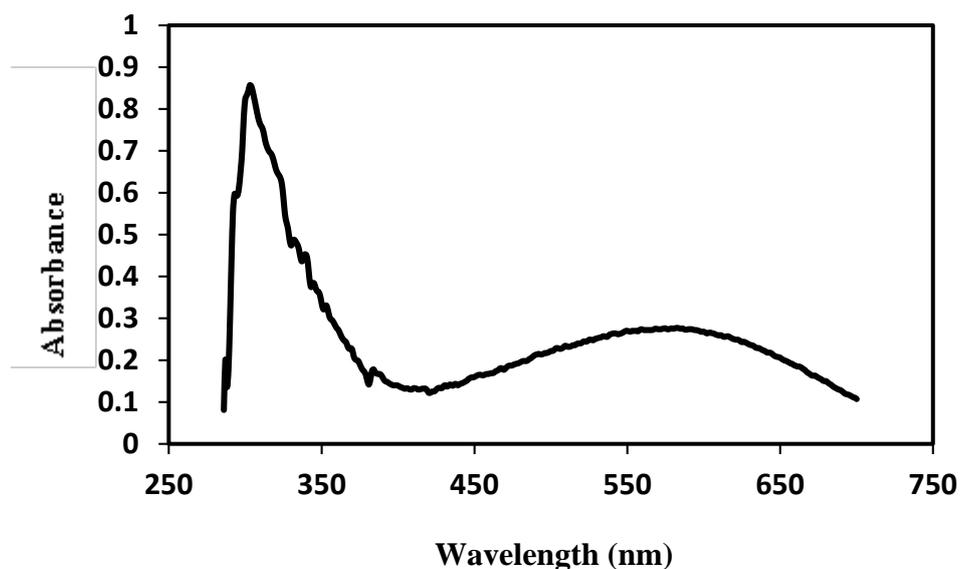
Fig. 3. UV–Vis spectra recorded for the poly(2-anisidine) coatingelectrosynthesized on the AA5052 electrode
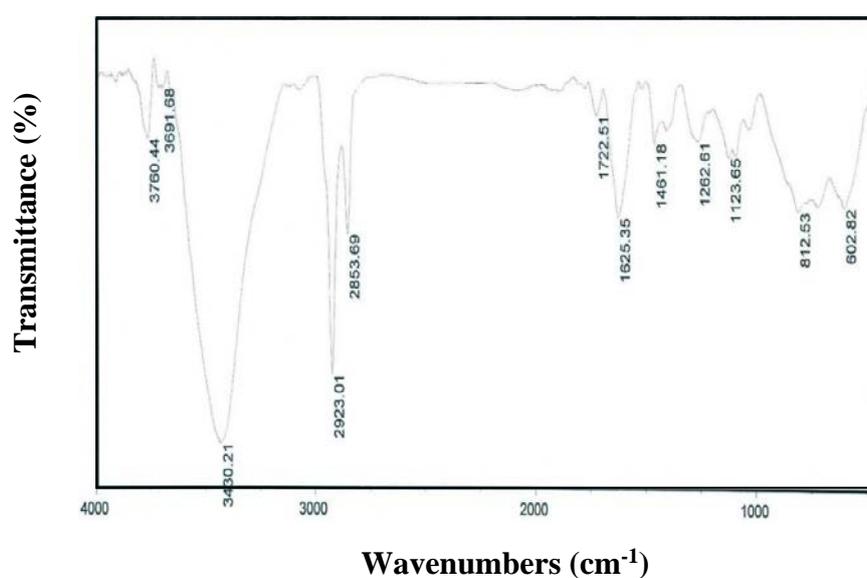
Fig. 4. FT-IR spectra of poly(2-anisidine) coating

3.3. Corrosion protection properties of the poly(2-anisidine) coating

The corrosion protection performance of the poly(2-anisidine) coatings synthesized on AA5052 was examinedat 3.5% NaCl solution using potentiodynamic polarization and EIS studies. The typical potentiodynamic polarization curves for uncoated AA5052 (bare) and poly(2-anisidine)-coated AA5052 electrode under galvanostatic conditions in an aqueous 3.5% NaCl solution, are shown in Fig. 5. Both the cathodic and anodic current decrease significantly when AA5052 electrode is coated with poly(2-anisidine). Poly(2-anisidine) coatings restrict the anodic and cathodic reactions of the electrode in the aggressive medium.

The values of E_{corr}, i_{corr}, corrosion rate (*CR*) and polarization resistance (R_p) (calculated using NOVA software) are given in Table 1. Analysis of these data shows that i_{corr} and the corrosion rate values decrease significantly when poly(2-anisidine) is galvanostaticallyelectrodeposited on AA5052.

From the measured i_{corr}values,the protection efficiency (*PE*) was obtained from the following equation [23]:

$$PE = \frac{i_{corr} - i_{corr(c)}}{i_{corr}} \times 100 \tag{1}$$

Where i_{corr} and $i_{corr(c)}$ are the values of the corrosion current density in theabsence and presence of poly(2-anisidine) coating, respectively.
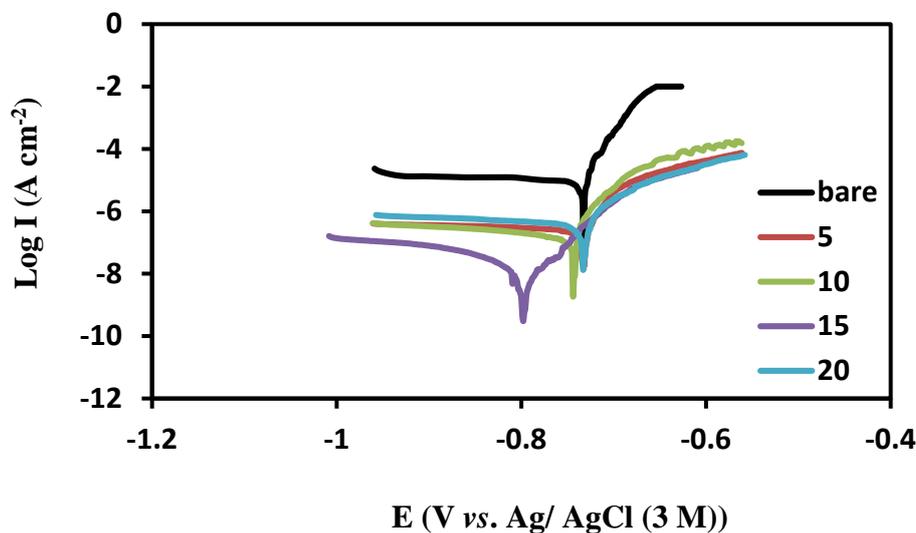

Fig. 5. The polarization behavior of poly(2-anisidine) coating electrosynthesized on AA5052 at various current densities (mA cm^{-2}) in 3.5% NaCl solution

As it can be seen in Table 1, i_{corr} values decrease from 5.338 µA cm^{-2} for uncoated AA5052 to 0.006 µA cm^{-2} for poly(2-anisidine)-coated AA5052 under optimal conditions (15 mA cm^{-2}). From Table 1, it also can be found that the corrosion rate (*CR*) of AA5052 is significantly reduced as a result of the reduction ofi_{corr}. The corrosion rate of the poly(2-

anisidine)-coated AA5052 is found to be 7.0×10^{-5} mm year^{-1}, which is ~900times lower than that observed for bare AA5052. The polarization resistance (R_P) is an important parameter, which shows the ability of a coating to prevent electron exchange in a corrosive environment. As it can be seen in Table 1, R_p increases from 0.76 kΩ cm^2 for uncoated AA5052 to 1765 kΩ cm^2 for poly(2-anisidine)-coated AA5052 under optimal condition. This shows that the poly(2-anisidine) coating has high polarization resistance. The *PE%* calculated from potentiodynamic polarization curve data is found to be 99.8% for the applied current density of 15 mA cm^{-2}.

Table 1. Electrochemical parameters of poly(2-anisidine) coatings electrosynthesizedon AA5052 in 3.5% NaClsolution under the galvanostatic conditions at different current densities for 1800 s

Sample	i_{corr} (μA cm^{-2})	E_{corr} (V vs. Ag/AgCl)	R_p (kΩ cm^2)	CR (mm year^{-1})	PE%	p%[*]
Bare	5.338	-0.732	0.76	6.2×10^{-2}	-	-
5 mA cm^{-2}	0.306	-0.730	36.27	3.5×10^{-3}	94.2	1.99
10 mA cm^{-2}	0.144	-0.741	46.79	1.7×10^{-3}	97.3	1.29
15 mA cm^{-2}	0.006	-0.796	1765	7.0×10^{-5}	99.8	0.0083
20 mA cm^{-2}	0.402	-0.728	27.80	4.7×10^{-3}	92.4	2.46

[*] (b_a: 0.089 V dec^{-1})

The coating porosity is one of the important parameters, which strongly governs the anti-corrosive behavior of the coatings. Therefore, measurement of the coating porosity is essential in order to estimate the overall corrosion resistance of the coated substrate. In this work, the porosity of poly(2-anisidine) coatings on AA5052 substrates was determined from potentiodynamic polarization measurements. The porosity of poly(2-anisidine) coating was calculated using the following relation [23]:

$$P = \frac{R_{puc}}{R_{pc}} \times 10^{-(\frac{|\Delta E|}{ba})} \qquad (2)$$

Where *P* is the total porosity, R_{puc} is the measured polarization resistance of the bare-AA5052, R_{pc} is the measured polarization resistance of the coated AA5052, ΔE_{corr} is the difference between corrosion potentials and b_a is the anodic Tafel slope for bare AA5052 substrate. The calculated porosity values of the poly(2-anisidine) coatings are also given in

Table 1. As seen in the table, the porosity value of the coating decreases with an increase in the applied current densities, indicating that the poly(2-anisidine) coating will deposit with better uniformity, regardless of the increase in the applied current density but this manner was reversed for the applied current density higher than 15 mA cm^{-2}.According to the results, the optimum current density for uniform deposit, low porosity and excellent corrosion resistance is 15 mA cm^{-2}.

In order to study the effect of applied current densities and used deposition times on the corrosion protection properties of the electrosynthesized coatings, four constant current densities of 5, 10, 15 and 20 mA cm^{-2} at different deposition times of 900, 1800, 2700 and 3600 s were used at the electropolymerization stage and the i_{corr} values obtained from the corresponding polarization curves are summarized in Table 2. As seen in the table, for all the applied current densities at the deposition times higher than 2700 s, the electrosynthesized coatings have the best corrosion protection properties (at the least i_{corr} values). On the other hand, for all the used deposition times by using the current densities more than 5 mA cm^{-2} and less than 20 mA cm^{-2}, the lowest i_{corr} values are observed.

Table 2. The i_{corr} (μA cm^{-2}) values obtained from polarization curves at different applied current densities for various deposition times

Applied current density	Deposition time (s)			
	900	1800	2700	3600
5 (mA cm^{-2})	3.15	0.306	0.031	0.098
10 (mA cm^{-2})	0.117	0.144	0.101	0.051
15 (mA cm^{-2})	3.3	0.006	0.004	0.075
20 (mA cm^{-2})	2.25	0.402	0.555	0.135

In this study, electrochemical impedance spectroscopy was also used to evaluate the corrosion protection efficiency for poly(2-anisidine)-coated AA5052. The typical Nyquist impedance plots of poly(2-anisidine)-coated AA5052 synthesized under galvanostatic conditions at different deposition times for 15 mA cm^{-2}recorded in 3.5% NaCl solution are shown in Fig. 6. These impedance plots were modeled by the equivalent circuit depicted in Fig. 7. The equivalent circuit consists the charge transfer resistance (R_{ct}), double layer capacitance (C_{dl}) and all of them are in series with the solution resistance (R_s). The R_{ct} is the charge transfer resistance of the area at the metal/coating interface at which corrosion occurs and C_{dl} is the corresponding capacitance.
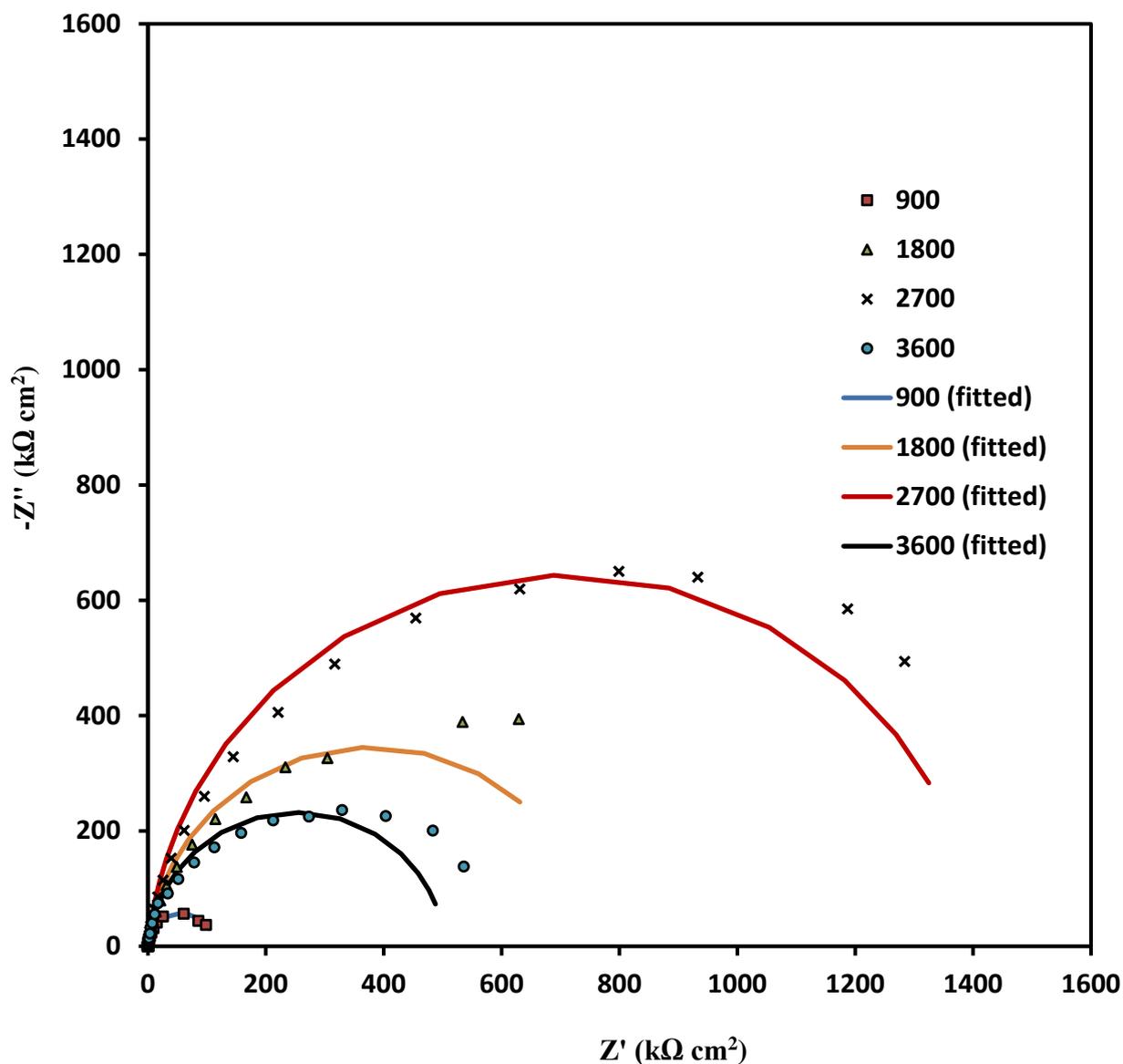
Fig. 6. Nyquist impedance plots for poly(2-anisidine)-coated AA5052 synthesized under galvanostatic conditions at various deposition times (s) for the applied current density of 15 mA cm^{-2}
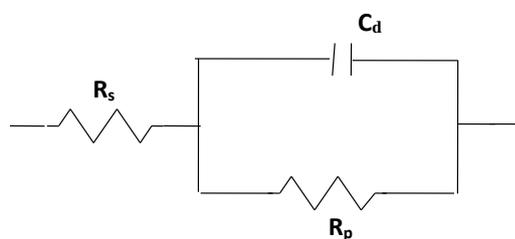

Fig.7. Equivalent circuit model

The values of the impedance parameters of the best fit to the experimental impedance plots for uncoated AA5052 and poly(2-anisidine)-coated AA5052 with the equivalent circuits

are given in Table 3. The protection efficiency (*PE*) was calculated using the following equation [23]:

$$PE = \frac{R_{ct(c)} - R_{ct}}{R_{ct(c)}} \times 100 \tag{3}$$

Where R_{ct} and $R_{ct(c)}$ are the charge transfer resistances in the absence and presence of the coating, respectively.

As seen in Table 3, the R_{ct} values increased and C_{dl} values decreased when the deposition time was increased up to 2700 s but this manner reversed when the deposition time was more than 2700 s. In other words, the most effective protection against corrosion was accomplished when the used deposition time was 2700 s, which confirms the obtained results of potentiodynamic polarization.

Table 3. Impedance parameter values of the poly(2-anisidine) coatings extracted from the fit to the equivalent circuit for the impedance spectra recorded in 3.5% NaCl solution as a function of the used deposition time

Sample	R_s (Ω cm^2)	R_{ct} (kΩ cm^2)	C_{dl} (μF cm^{-2})	*PE%*
Bare	4.4	4	10.7	-
900 s	8.8	133	1.12	96.9
1800 s	7.2	865	1.16	99.5
2700 s	6.6	1410	1.15	99.7
3600 s	6.0	526	1.18	99.2

The R_{ct} value is 1410 kΩ cm^2 (under optimal conditions), which is about 352 times higher than that of uncoated AA5052. The higher value of R_{ct} is attributed to the effective barrier behavior of the poly(2-anisidine) coating. The lower values of C_{dl} for the poly(2-anisidine)-coated AA5052 provide further support for the protection of AA5052 by the poly(2-anisidine) coating. A uniform and defect-free coated metal is characterized by higher R_{ct} and lower C_{dl}. Higher values of R_{ct} and the lower values of C_{dl} indicate that the coatings offer excellent corrosion performance for AA5052. The *PE%* calculated from EIS data is found to be 99.7%, which is in agreement with the potentiodynamic polarization results. The R_{ct} and

PE% calculated from EIS data are found that these values decrease with increasing applied current density, which is in agreement with the potentiodynamic polarization results.

The R_{ct} values obtained from Nyquist impedance plots for all applied constant current densities at different deposition times are summarized in Table 4. As seen in the table, which is in agreement with the potentiodynamic polarization results, for all the applied current densities at the deposition times higher than 2700 s, the electrosynthesized coatings have the best corrosion protection properties (with the maximum R_{ct} values). On the other hand, for all the used deposition times by using the current densities more than 5 mA cm^{-2} and less than 20 mA cm^{-2}, the highest R_{ct} values are observed.

Table 4. The R_{ct} (kΩ cm^2) values obtained from Nyquist curves at different applied current densities for various deposition times

Applied current density	Deposition time (s)			
	900	1800	2700	3600
5 (mA cm^{-2})	68	308	960	532
10 (mA cm^{-2})	195	165	262	548
15 (mA cm^{-2})	133	865	1410	526
20 (mA cm^{-2})	157	241	67	459

3.4. SEM characterization

Fig. 8 shows the typical SEM images of abraded AA5052 (image a), prepared AA5052 after corrosion(image b), poly(2-anisidine) coating grown by an applied current density of 15 mA cm^{-2} for 2700 s (image c) and the poly(2-anisidine)-coated AA5052 after corrosion(image d). A comparison of images (a) and (b) shows that numerous large pits and inequalities were formed after corrosion, which reveals severe damage on the surface due to metal dissolution. Image c shows that the poly(2-anisidine) coating was uniformly electrodeposited on the surface. A comparison of images c and d shows that the poly(2-anisidine) coating protects the AA5052 from the corrosion, which does not change dramatically.
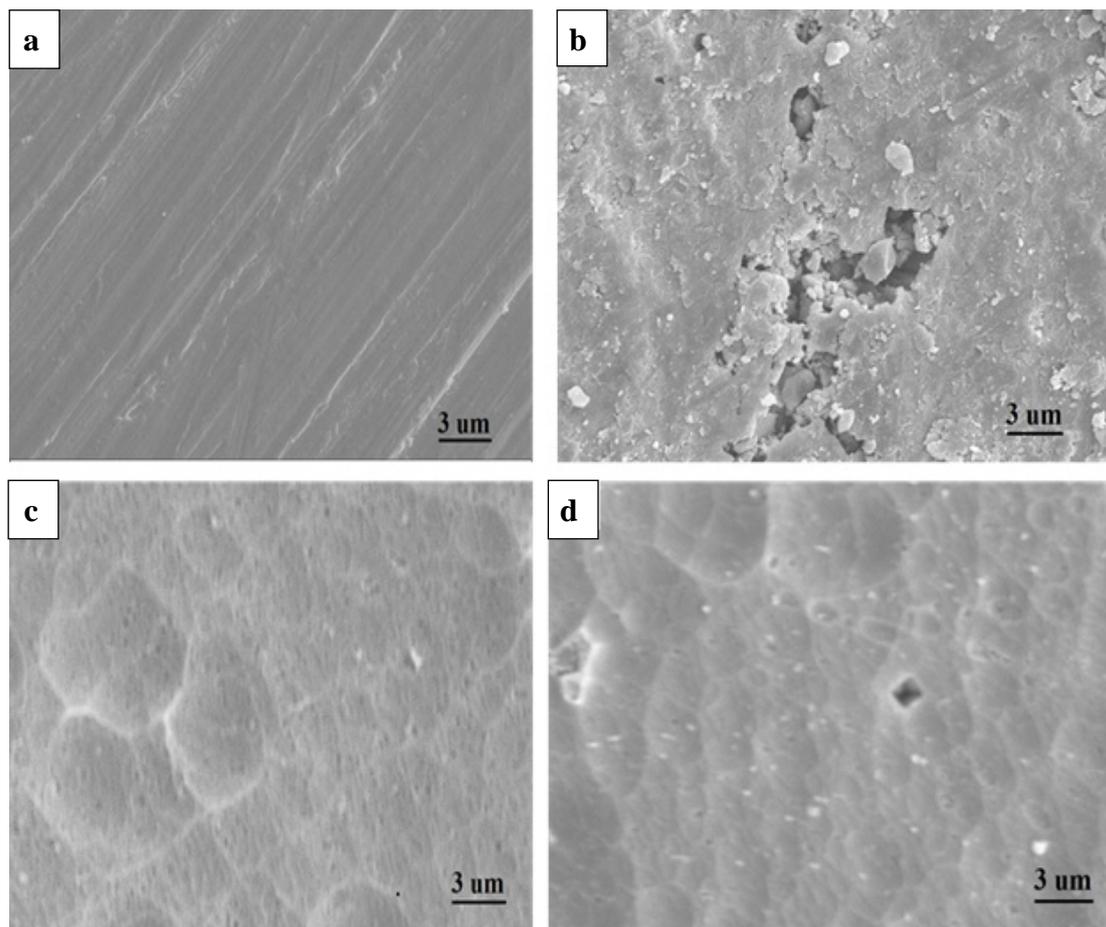
Fig.8. SEM micrographs of abraded AA5052 (image a) pre-treated AA5052 after 3 hours immersion in corrosive environments at room temperature (image b), poly(2-anisidine) coating grown by applying current density of 15 mA cm^{-2} to 2700 son AA5052 electrode (c) and poly(2-anisidine) coating after 3 hours immersion in corrosive environments at room temperature(d)

4. CONCLUSIONS

Poly(2-anisidine) coatings were successfully electrosynthesized on AA5052 substrates from aqueous solutions containing oxalic acid and 2-anisidine monomers. Uniform, compact and strongly adherent coatings can be obtained under galvanostatic conditions. Four different constant current densities of 5, 10, 15 and 20 mA cm^{-2} at different deposition times of 900, 1800, 2700 and 3600 s were applied to the formation of poly(2-anisidine) coatings on AA5052. The results showed that the current density of 15 mA cm^{-2} at the deposition time of 2700 s for the polymerization stage is the best conditions for the synthesis of more compact and strongly adherent poly(2-anisidine) coatings on AA5052. The green coatings, characteristic of poly(2-anisidine) in the emeraldine oxidation state, were produced on the surface of AA5052 using galvanostatic technique. The poly(2-anisidine) coatings were characterized by FT-IR, UV-Vis and SEM techniques and the corrosion protection properties

of the electropolymerized coatings were evaluated using Tafel polarization and electrochemical impedance spectroscopy in 3.5% NaCl solution. According to the results, the optimum applied current density for uniform deposit, low porosity and excellent corrosion resistance was 15 mA cm^{-2}. The EIS results are in good agreement with the potentiodynamic polarization measurements. This study reveals the poly(2-anisidine) coating has excellent corrosion protection properties and can be considered as a potential coating material to protect AA5052 against corrosion in aqueous chloride-containing solutions.

Acknowledgement

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