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The Effect of Anodization and Subsequent Treatments on Corrosion Resistance of Aluminium

ABSTRACT

Aluminium samples were chemically prepared by following operations: degreasing, etching I, etching II, and brightening), prior to anodizing in sulfuric acid. Aluminium surface area was 0.2 dm². The composition of used aluminium samples was determined by the energy dispersive X-ray spectroscopy. Chemically prepared aluminium samples were electrochemically anodized for 45 minutes in a solution of 190 gdm⁻³ H₂SO₄ at room temperature, at a current density of 1.7 Adm⁻². In the anodizing process, the aluminium sample served as the anode, with lead cathodes. After anodizing, the aluminium samples underwent a colouring process in five pairs of solutions (systems), where each system consisted of two solutions of inorganic salts RxA + RxB (x = 1-5, numbers of solutions). Colouring of the anodized aluminium was carried out at room temperature by immersing the samples in each solution for 7 minutes (e.g., R1A + R1B, τ=7 min + 7 min). Each used colouring system provides a different colour: green-yellow, brown, light-grey, blue, and orange-gold. After colouring, the samples were treated in a special solution to improve corrosion resistance and silication, resulting in a change in the obtained colour shade. All obtained colours were stable with very nice appearance, allowing such coloured aluminium to be used for decorative purposes. The corrosion resistance of the coloured anodized aluminium samples was investigated by determining the corrosion potential, corrosion current and polarization resistance using potentiodynamic polarization method, as well as by electrochemical impedance spectroscopy. A common feature of all tested samples is a significant improvement in the corrosion resistance of the anodized aluminium after colouring and subsequent treatment in the corrosion resistance improvement solution, particularly after the additional silane treatment.

Keywords: Aluminium anodization, electrochemical impedance spectroscopy, potentiodynamic polarization, chemical colouring, silication, corrosion resistance.

1. INTRODUCTION

Aluminium is the third most abundant element in the Earth's crust, following oxygen and silicon, and exhibits a range of significant properties. The high mechanical strength of some of its alloys at low specific weight, good electrical and thermal conductivity, corrosion resistance, and various other characteristics make it widely applicable. Primarily used in aviation and military industries, aluminium and its alloys

are now prominent in electronics, chemistry, petroleum refining, machinery, construction, and automotive industries [1-3].

Reaction of aluminium and oxygen in atmospheric conditions, forming an oxide layer is one of the most important characteristic of aluminium. The excellent chemical stability of aluminium can be attributed to the presence of a passive oxide film [4-7]. Experimental measurements of the corrosion rate of high-purity aluminium in solutions with different pH values indicate that the corrosion rate in neutral solutions is very low (a few μAcm⁻²). A significant increase in the corrosion rate occurs in acidic and alkaline solutions, with a higher increase observed in alkaline solutions. The process of

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anodic oxidation (anodization) on the surface of aluminium produces an oxide coating with enhanced protective (anticorrosive, anti-abrasive), electro-insulating, and adsorptive properties. This expands the application possibilities of aluminium and its alloys. The proper combination of operating parameters such as temperature, electrolyte type, applied voltage, current density, and duration of anodization are crucial to obtain an oxide film with advanced characteristics. Anodic coatings can be formed on aluminium using a variety of formulated electrolytes with the application of either direct or alternating current. Due to the porosity of the oxide layer, special attention is required for the final treatment of samples to close the pores in the coating and enhance corrosion resistance. Final treatment typically involves rinsing, colouring, and sealing. The oxide film formed on aluminium has good adsorption properties, allowing it to be coloured in various ways, including the use of organic or inorganic substances and electrochemical colouring (direct or alternating current) [8-18]. The colouring process not

only improves corrosion resistance but also provides a decorative effect, significantly expanding the application areas of aluminium.

The aim of this study is to investigate the corrosion resistance of aluminium after each processing stage: chemical preparation, anodization of aluminium in sulfuric acid, chemical colouring of aluminium in different solutions, and subsequent treatment to enhance corrosion resistance. Additionally, the goal was to obtain a final product (aluminium) in various colours and shades that, along with improved corrosion resistance, possesses enhanced decorative properties. This collectively broadens the possibilities of using aluminium as a structural material in various industries.

EXPERIMENTAL PART

Aluminium samples with a surface area of 0.2 dm², whose composition was determined using the Shimadzu EDX-8000 device and is presented in Table 1, were used for experiments.

Table 1. Composition of the aluminium sample expressed in wt. %

Element	Al	Fe	P	Ta	K	Ca	Zn	Sm
Wt.%	98.899	0.267	0.197	0.118	0.109	0.109	0.064	0.059
Element	Ba	Mn	Cr	V	Ga	As	Ni	Tl
Wt.%	0.054	0.036	0.029	0.026	0.014	0.012	0.006	0.003

Aluminium samples were subjected to a chemical preparation process before anodization in sulfuric acid: degreasing, etching I, etching II, and brightening. Degreasing was carried out in a solution: 50% Na₂CO₃, 50% Na₃PO₄·10H₂O, t= 60-70°C, τ= 15 min., 5% water solution, pH= 7.5 – 8.5. Etching I was performed in a solution: 20% NaOH, 2% NaF, τ=2-5 min., t= 70-75°C, and etching II in a solution: 550 cm³dm⁻³ HNO₃, 200 cm³dm⁻³ H₂SO₄, 140 cm³dm⁻³ NaF, τ= 5 min., at room temperature. Brightening was carried out in a solution: 20% HNO₃, 10% HF, τ= 5 min., at room temperature. After chemical preparation, the samples were subjected to anodization process in a solution of 190 gdm⁻³ H₂SO₄, at room temperature, τ= 5 min., and at a current density of 1.7 Adm⁻². After anodization, the aluminium samples underwent a chemical staining process in five different colouring systems, at room temperature. Each system consists of two solutions of inorganic salts, and the colouring time was the same in each of the

solutions (τ=7 min. + 7 min.). The following solutions were used [19]:

1. Solution for achieving yellow colour (R1A + R1B): 50-100 gdm⁻³ K₂Cr₂O₇ + 100-200gdm⁻³ Pb(CH₃COO)₂.
2. Solution for achieving brown colour (R2A + R2B): 10-50 gdm⁻³ K₄[Fe(CN)₆]·3H₂O + 10-100gdm⁻³ CuSO₄.
3. Solution for achieving light grey colour (R3A + R3B):, 10-50 gdm⁻³ Pb(CH₃COO)₂ + 10-50 gdm⁻³ Na₂SO₄.
4. Solution for achieving blue colour (R4A + R4B): 10-15 gdm⁻³ K₄[Fe(CN)₆]·3H₂O + 10-100 gdm⁻³ FeCl₃.
5. Solution for achieving golden yellow colour (R5A + R5B): 10-50 gdm⁻³ Na₂S₂O₃ + 10-50 gdm⁻³ KMnO₄.

After staining, the aluminium samples were treated in a solution of following composition, to improve

corrosion resistance (R_6) [19]: $0.1 \text{ gdm}^{-3} \text{ CoSO}_4$, $5.5 \text{ gdm}^{-3} \text{ CH}_3\text{COONa}$, $3.5 \text{ gdm}^{-3} \text{ H}_3\text{BO}_3$, $\text{pH} = 4.7\text{-}5.7$, at a temperature of $80\text{-}85^\circ\text{C}$ or by immersion for 3 min, in a solution of hot water at $t = 98^\circ\text{C}$ (silane treatment).

Surface of the aluminium samples were observed by Leica EZ4 HD optical microscope. The effect of treatments listed above on the corrosion resistance of aluminium in 3%NaCl was examined by potentiodynamic polarization method and electrochemical impedance spectroscopy. This corrosion measurements were performed using a potentiostat/galvanostat/ZRA Gamry Series GTM 750, in a standard three-electrode cell. The saturated calomel electrode was used as a reference electrode, a Pt electrode was used as a counter electrode and treated aluminium sample was working electrode. Tested sample surface area was 1 cm^2 . The Tafel plots was recorded in the range of $\pm 0.25 \text{ V}$ with respect to the open circuit potential, with a recording speed of 1 mV/s . Electrochemical impedance spectroscopy frequency range was $100\text{kHz} - 10\text{mHz}$, with a 7 mV sinusoidal voltage amplitude, at open circuit potential. The corrosion current density, corrosion potential, cathodic and anodic slope were calculated based on Tafel plots by the DC Corrosion Software for ZRA Gamry Series GTM 750 potentiostat/galvanostat.

The polarization resistance, R_p (Ωcm^2), is calculated according to Stern-Geary equation:

$$R_p = \frac{\beta_a \beta_c}{2.3 j_{\text{corr}} (\beta_c + \beta_a)} \quad (1)$$

Where $\beta_a \beta_a$ and $\beta_c \beta_c$ are anodic and cathodic Tafel slope in V, j_{corr} is corrosion current density in A/cm^2 .

RESULTS AND DISCUSSION

The aluminium samples surfaces were analysed before and after colouring process, and after subsequent silane treatment or immersion in a solution for improving corrosion resistance. Photographs of aluminium surfaces are given in Figures 1-6.

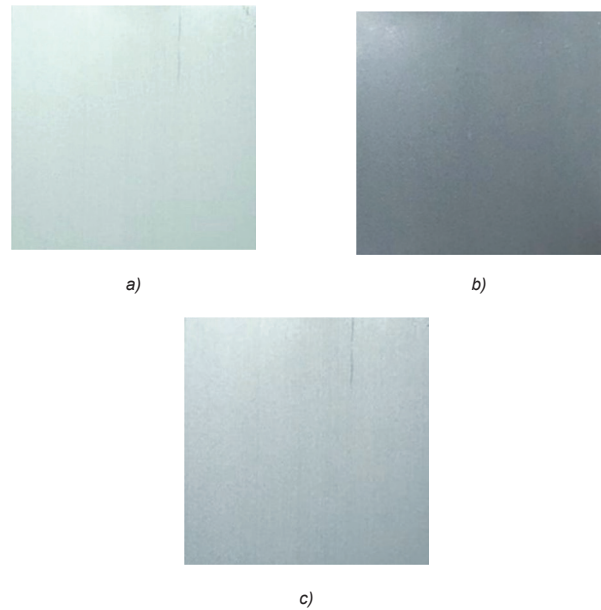


Fig. 1. Photographs of aluminium surface after: a) chemical preparation; b) anodization; c) silane treatment

From Figure 1, it can be observed that the aluminium sample after chemical preparation has a shiny silver colour, which turns into a darker matte colour during anodization in sulfuric acid. After subsequent treatment by silication, it remains a lighter shade similar to that of the chemically prepared sample.

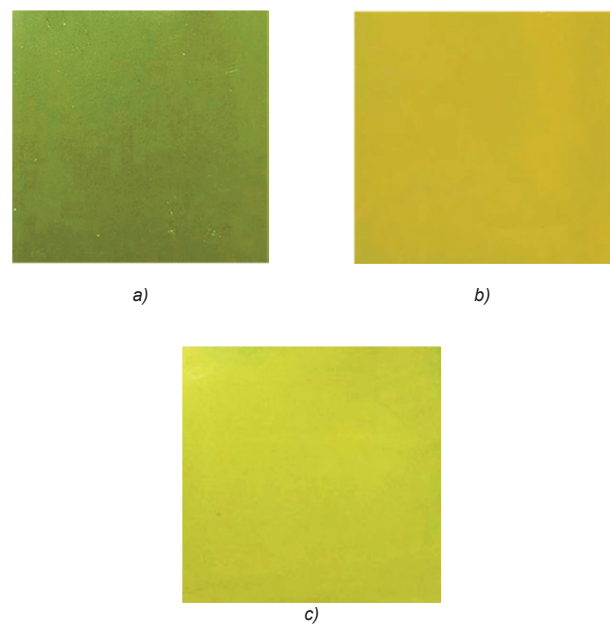


Fig. 2. Photographs of anodized aluminium surface: a) coloured in solutions R1A+R1B; b) after silane treatment; c) after treatment in solution R6

It can be seen from Figure 2 that the colour of the aluminium sample changes significantly after colouring with solutions R1A and R1B, from greenish-yellow to a darker yellow. After the silane treatment, the colour changed to a light yellow-green colour, and after treatment in solution R6, it becomes a light yellow-green shade.

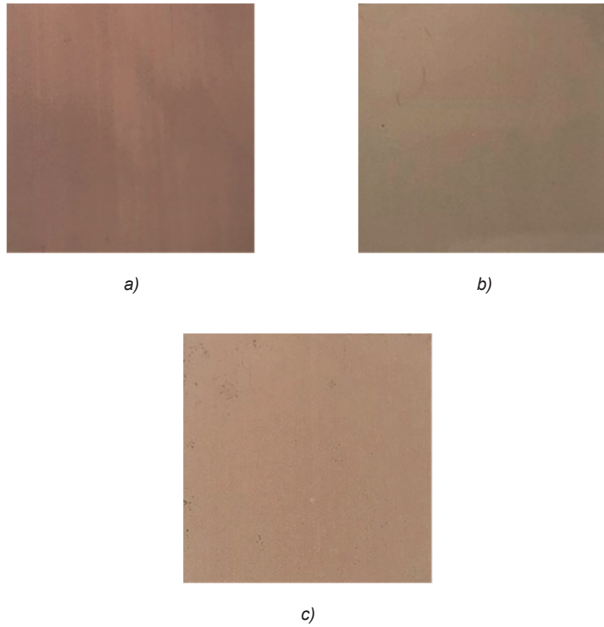


Fig. 3. Photographs of anodized aluminium: a) coloured in solutions R2A+R2B; b) after silane treatment; c) after treatment in solution R6

The surface of anodized aluminium after colouring with solutions R2A+R2B exhibits a brown colour. After subsequent silane treatment and treatment in solution R6, there is a slight change in the shade of brown colour, as seen in Figure 3.

Chemically coloured anodized aluminium in solutions R3A+R3B, and subsequent silane treatment or treatment in solution R6, results in different shades of light grey colour, as shown in Figure 4.

The anodized aluminium samples chemically coloured in solutions R4A+R4B display an intense blue colour, as shown in Figure 5. After subsequent silane treatment or treatment in the solution for improving corrosion resistance, there is a slight change in colour. After treatment in solution R6, the surface of the sample turns into a matte blue.

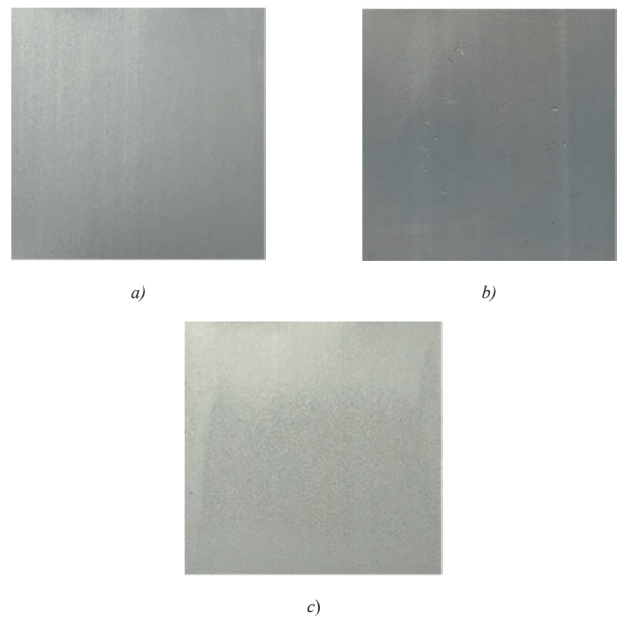


Fig. 4. Photographs of anodized aluminium: a) coloured in solutions R3A+R3B; b) after silane treatment; c) after treatment in solution R6

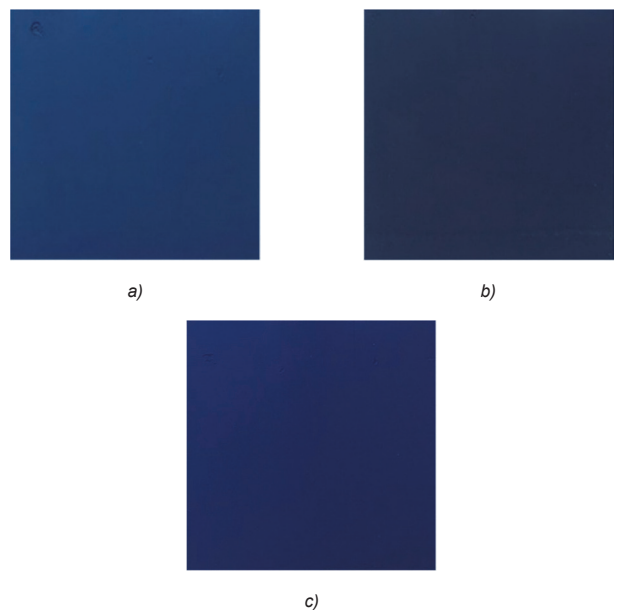


Fig. 5. Photographs of anodized aluminium: a) coloured in solutions R4A+R4B; b) after silane treatment; c) after treatment in solution R6

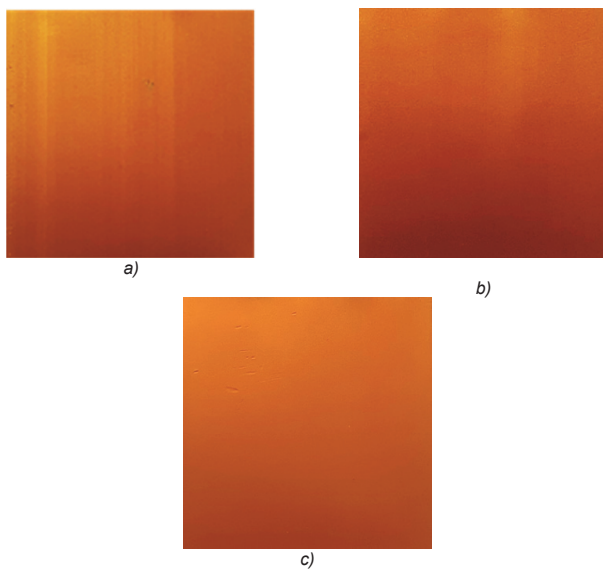


Fig. 6. Photographs of anodized aluminium: a) coloured in solutions R5A+R5B; b) after silane treatment; c) after treatment in solution R6

It can be observed from Figure 6 that aluminium samples chemically coloured in solutions R5A+R5B

exhibit an intense orange-gold colour. After treatment in solution R6, the surface of the sample retains its intense orange-gold colour.

Figures 1-6 demonstrate that through chemical treatment in various inorganic solutions, anodized aluminium can be coloured in different shades, expanding its potential utilization as a structural material for interior and exterior decoration, as well as for creating devices with advanced appearances. Corrosion resistance of aluminium after chemical preparation, anodization, colouring, and subsequent silane treatment by or treatment in a solution for improving corrosion resistance was examined by potentiodynamic polarization method and by electrochemical impedance spectroscopy (EIS). Figures 7–11 depict Tafel polarization diagrams showing the potential (E) dependence on the logarithm of current density (log j). The relevant electrochemical parameters obtained from the polarization curves, including corrosion potential (E_{corr}), corrosion current density (j_{corr}), corrosion rate (π) and polarization resistance (R_p) are shown in Table 2.

Aluminium after:	j_{corr} ($nAcm^{-2}$)	$-E_{corr}$ (V)	π ($\mu mgod^{-1}$)	R_p ($M\Omega cm^2$)
Chemical preparation	3890	0.81	130	0.008
Anodization	21.10	0.58	0.70	4.80
R1A+R1B	111.1	0.68	30.0	0.120
R1A+R1B+silane treatment	0.548	0.59	0.02	56.0
R1A+R1B+R6	1.630	0.66	0.05	21.0
R2A+R2B	49.90	0.59	1.60	8.80
R2A+R2B+silane treatment	0.649	0.57	0.03	33.0
R2A+R2B+R6	35.10	0.64	1.20	3.80
R3A+R3B	1700	0.67	57.0	0.003
R3A+R3B+silane treatment	72.70	0.58	2.30	0.30
R3A+R3B+R6	31.90	0.62	1.10	1.50
R4A+R4B	1600	0.66	54.0	0.04
R4A+R4B+silane treatment	0.481	0.54	0.02	9.00
R4A+R4B+R6	21.10	0.61	0.70	2.00
R5A+R5B	4.900	0.50	0.16	8.70
R5A+R5B+silane treatment	1.890	0.69	0.25	23.0
R5A+R5B+R6	1.610	0.60	0.05	21.0

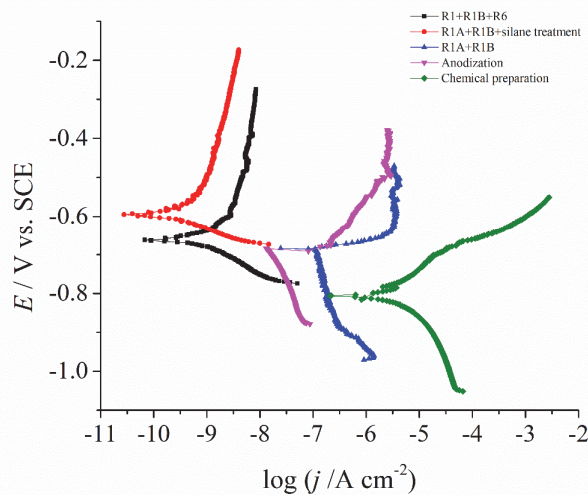


Fig. 7 Tafel plots for aluminium after chemical preparation, anodization, colouring in R1A+R1B, subsequent silane treatment, and treatment in solution R6

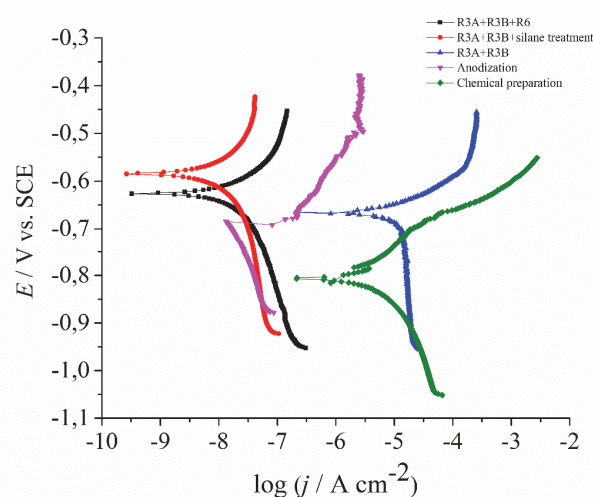


Fig. 9 Tafel plots for aluminium after chemical preparation, anodization, colouring in R3A+R3B, subsequent silane treatment, and treatment in solution R6

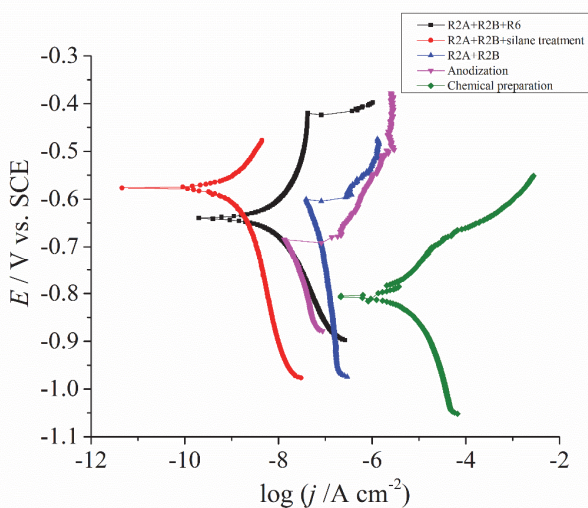


Fig. 8 Tafel plots for aluminium after chemical preparation, anodization, colouring in R2A+R2B, subsequent silane treatment, and treatment in solution R6

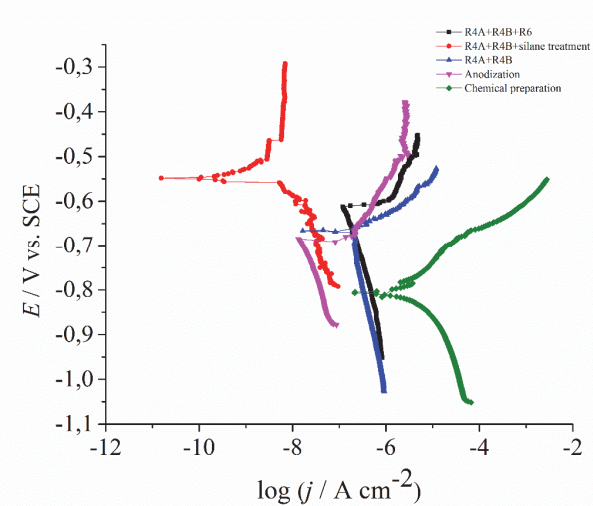


Fig. 10 Tafel plots for aluminium after chemical preparation, anodization, colouring in R4A+R4B, subsequent silane treatment, and treatment in solution R6

From the Figures 7-11 and from the Table 2 it is evident that all examined treatments, i.e. anodization, colouring and silane treatment improves the corrosion resistance of aluminium in saline environment compared to chemical treated aluminium sample. The corrosion current is decreased more than 5 thousand times only by anodization, and by further treatments like colouring and silane treatment the corrosion current value changed from μAcm^{-2} to pAcm^{-2} . The most corrosion resistant aluminium

sample was after colouring and silane treatment, which implies the importance of pore closure for good aluminium corrosion stability. The silane treatment showed better results compared to examine solution for enhancement corrosion resistance (R6). From the results of corrosion rate and polarization resistance it can be seen that aluminium samples coloured in solutions R5A+R5B exhibit the best corrosion resistance.

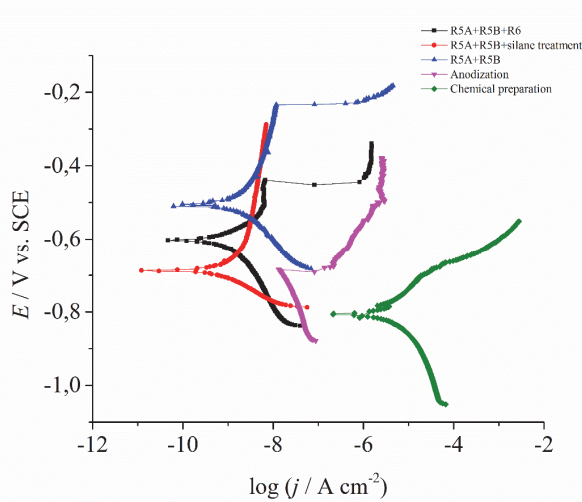
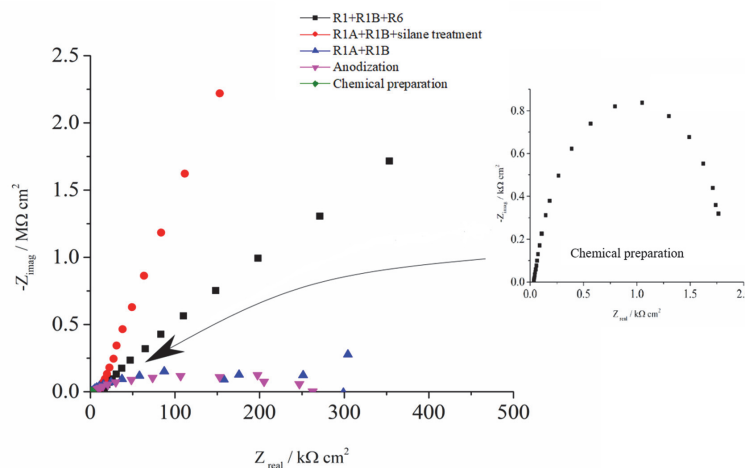
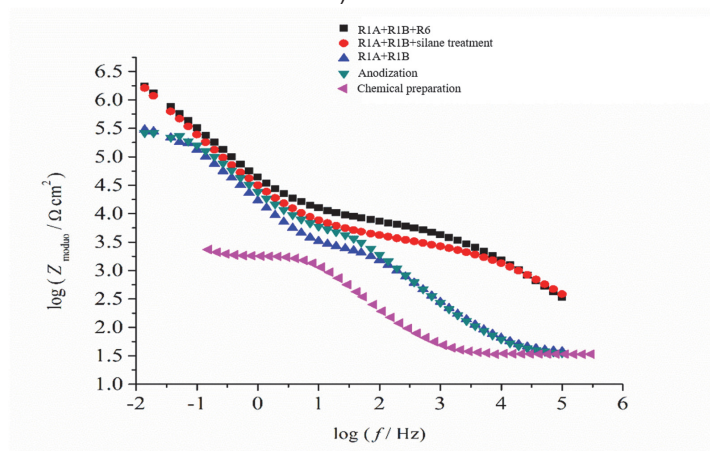


Fig. 11 Tafel plots for aluminium after chemical preparation, anodization, colouring in R5A+R5B, subsequent silane treatment, and treatment in solution R6

In general, the colouring process increases corrosion resistance of anodized aluminium. The best corrosion resistance, i.e., the most positive corrosion potential, is exhibited by aluminium coloured in solutions R5A+R5B ($E_{corr} = -0.50V$), and no subsequent treatment is needed after colouring in these solutions since corrosion parameters are not changing significantly by further treatments. After colouring of anodized aluminium in solutions (R1A+R1B; R2A+R2B; R3A+R3B; and R4A+R4B), the subsequent treatment in solution R6, and especially silication, significantly improves corrosion resistance. Silane treatment proves to be very effective, as it not only enhances the corrosion resistance of coloured samples but also has no significant impact on the colour change before and after treatment.



a)



b)

Fig. 12. a) Nyquist plot and b) Bode plot for aluminium after chemical preparation, anodization, colouring in R1A+R1B, subsequent silane treatment, and treatment in solution R6

Figures 12–17 show graphs depicting the dependence of $-Z_{imag}$ on Z_{real} (Nyquist plots) and the dependence of $\log Z_{moduo}$ on $\log f$ (Bode plots) obtained by electrochemical impedance spectroscopy (EIS) for aluminium samples that were chemically prepared, anodized, coloured in solutions (R1A+R1B; R2A+R2B; R3A+R3B; R4A+R4B; and R5A+R5B), subsequently treated in solution R6, and further treated by silication.

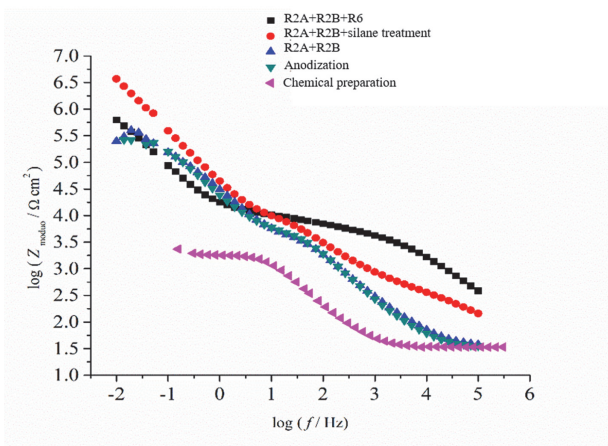
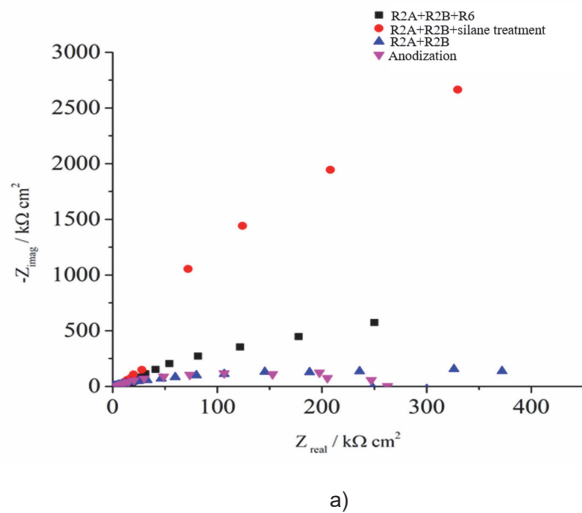


Fig. 13. a) Nyquist plot and b) Bode plot for aluminium after chemical preparation, anodization, colouring in R2A+R2B, subsequent silane treatment, and treatment in solution R6

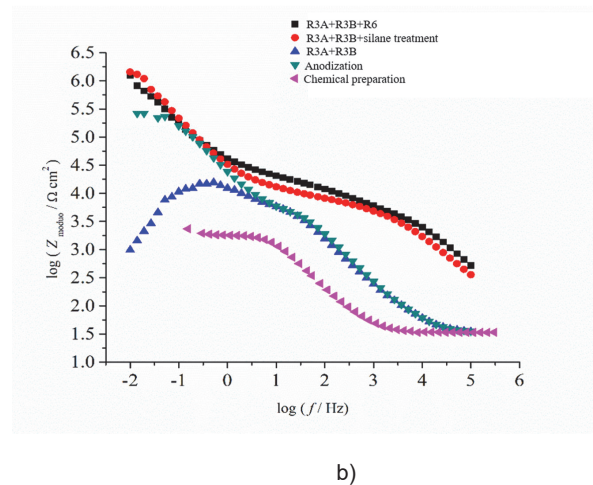
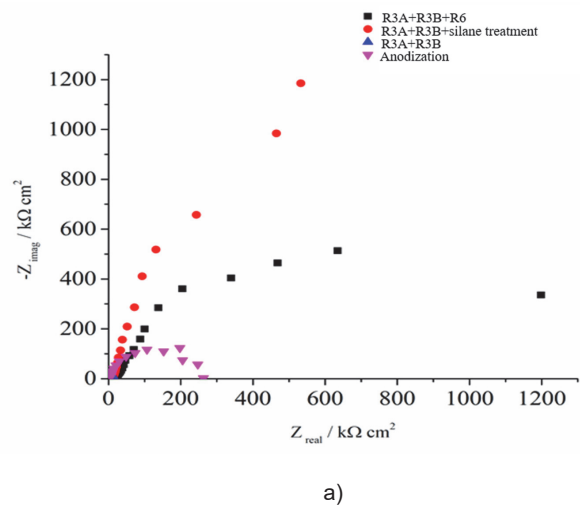


Fig. 14. a) Nyquist plot and b) Bode plot for aluminium after chemical preparation, anodization, colouring in R3A+R3B, subsequent silane treatment, and treatment in solution R6

Based on the Nyquist and Bode plots (Figures 12–15), it can be generally stated that coloured anodized aluminium in solutions (R1A+R1B; R2A+R2B; R3A+R3B; and R4A+R4B), subsequently treated in solution R6, and especially silication, significantly improve corrosion resistance. Moreover, all samples treated by silication exhibit the highest corrosion resistance.

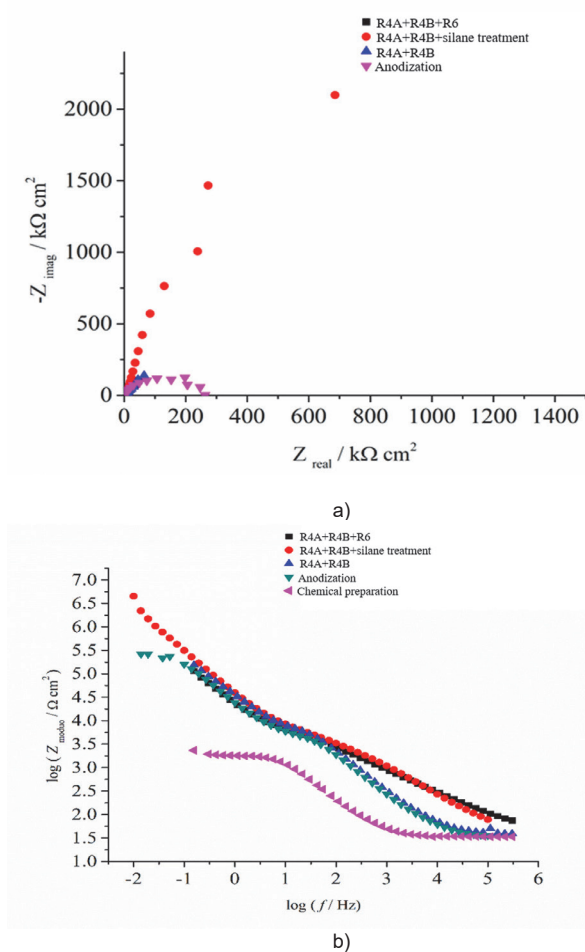


Fig. 15. a) Nyquist plot and b) Bode plot for aluminium after chemical preparation, anodization, colouring in R4A+R4B, subsequent silane treatment, and treatment in solution R6

From the Nyquist and Bode plots (Figure 16), it is evident that aluminium samples coloured after anodization in solutions R5A and R5B demonstrate the highest corrosion resistance. Aluminium samples coloured in solutions R5A and R5B and subsequently treated in solution R6, as well as those treated by silication, show lower resistance to corrosion. This indicates that aluminium painted in R5A and R5B does not require subsequent treatment.

The results obtained from the Tafel polarization diagrams and corrosion resistance of the examined aluminium samples are in complete agreement with the results from electrochemical impedance spectroscopy (EIS). Anodized aluminium coloured in solutions (R1A+R1B; R2A+R2B; R3A+R3B; and R4A+R4B), after subsequent treatment in solution R6, exhibits improved corrosion resistance, while the

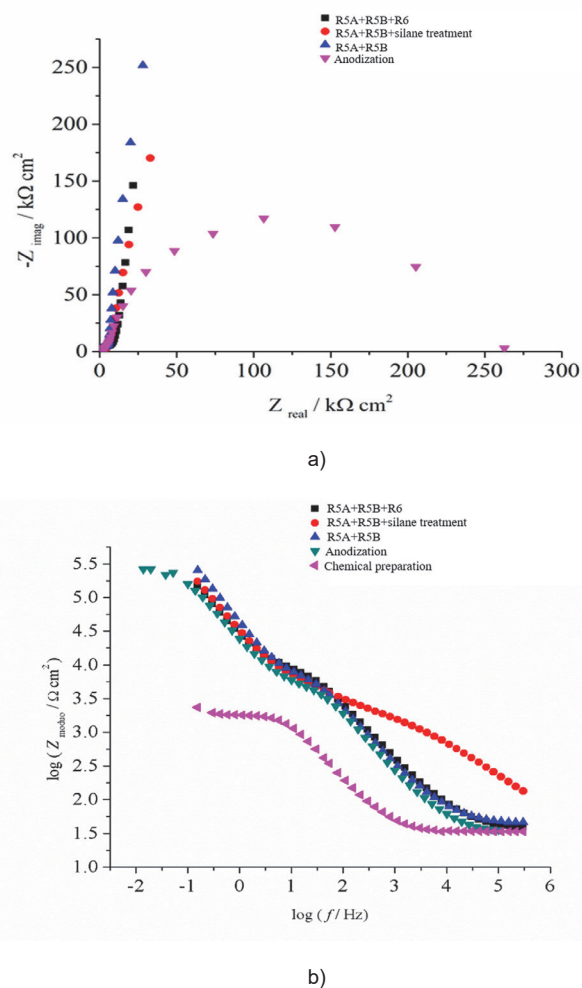


Fig. 16. a) Nyquist plot and b) Bode plot for aluminium after chemical preparation, anodization, colouring in R5A+R5B, subsequent silane treatment, and treatment in solution R6

highest corrosion resistance is observed in all samples treated by silication.

CONCLUSION

Anodizing aluminium in sulfuric acid under defined conditions forms an oxide layer with specific characteristics that significantly influence the corrosion resistance of the base metal. By chemically colouring anodized aluminium and subsequent treatments, either with solution R6 or silication, a final product (aluminium) of various colours and shades with improved corrosion resistance is obtained. By chemical colouring of anodized aluminium in the proposed combination of solutions it is possible to obtain the green-yellow, brown colour, light grey, blue, and orange-gold colour. Subsequent treatment

by silication minimally affects shade of colour of the chemically coloured sample, while solution R6, in addition to a slight change in colour shade, mattifies the surfaces. Results gained by potentiodynamic polarization and electrochemical impedance spectroscopy methods showed that colouring of anodized samples in all used pairs of solutions increases corrosion resistance. By applying subsequent treatment in solution R6, and especially silication, corrosion resistance is increased, except for anodized aluminium coloured in solutions R5A+R5B, which had very good corrosion resistance in 3%NaCl after chemical colouring and did not require subsequent treatment. This indicates the reliability of the obtained results and the methods used.

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IZVOD

UTICAJ ANODIZACIJE I NAKNADNIH TRETMANA NA OTPORNOST ALUMINIJUMA NA KOROZIJU

Uzorci aluminijuma površine $0,2 \text{ dm}^2$ pre anodizacije u sumpornoj kiselini podvrgnuti su procesu hemijske pripreme (odmašćivanje, nagrivanje I, nagrivanje II i odmašćivanje). Sastav uzoraka aluminijuma određen je energetsom disperzijom X-zraka na uređaju Shimadzu EDX-8000. Tako pripremljeni uzorci aluminijuma su elektrohemijski anodizovani 45 minuta u rastvoru $190 \text{ gdm}^{-3} \text{ H}_2\text{SO}_4$ na sobnoj temperaturi, pri gustini struje $1,7 \text{ Adm}^{-2}$. U procesu anodizacije kao anoda korišćen je uzorak aluminijuma, a katode su bile od olova. Nakon anodizacije uzorci aluminijuma su podvrgnuti procesu bojenja u pet parova rastvora (sistema), gde se jedan sistem sastoji od dva rastvora neorganskih soli $R_xA + R_xB$ ($x = 1-5$, broj rastvora). Bojenje anodizovanog aluminijuma vršeno je na sobnoj temperaturi potapanjem uzoraka u svaki rastvor po 7 minuta (npr. R1A + R1B, $\tau = 7 \text{ min} + 7 \text{ min}$). Svaki korišćen sistem za bojenje aluminijuma daje različitu boju: zeleno-žutu, braon, svetlo-sivu, plavu i narandžasto-zlatnu boju. Nakon bojenja uzorci su obrađivani u rastvoru za poboljšanje korozione postojanosti i siliranjem, usled čega dolazi do promene dobijene nijanse boje. Sve dobijene boje su stabilne i dekorativne, tako da se ovako obojeni aluminijum može koristiti i u dekorativne svrhe.

Koroziona postojanost obojenih anodiziranih uzoraka aluminijuma ispitivana je određivanjem korozionog potencijala i struje korozije pomoću snimljenih polarizacionih dijagrama, kao i pomoću spektroskopije elektrohemijske impedanse (SIE).

Zajedničko svim ispitivanim uzorcima je značajno poboljšanje korozione postojanosti anodno obrađenog aluminijuma nakon bojenja i naknadne obrade u rastvoru za poboljšanje korozione postojanosti, a posebno nakon naknadne obrade siliranjem

Ključne reči: Anodizacija aluminijuma, spektroskopija elektrohemijske impedanse, polarizacioni dijagrami, hemijsko bojenje, siliranje, koroziona postojanost.

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