

The utilization of metal materials finds widespread applications in various industries, including the aircraft industry, where aluminum alloys are commonly employed. However, metal materials are prone to corrosion under specific conditions, necessitating the implementation of corrosion prevention methods to decelerate the material's corrosion rate. Corrosion is a process in which the quality of metal deteriorates due to environmental influences. An effective approach to inhibit corrosion is through anodizing, which involves applying a protective coating to the metal surface, preventing direct contact with the surrounding environment. In this research, the focus was on studying the corrosion rate of aluminum alloy 2024 using Boric Sulfate Acid Anodizing (BSAA) at 10 volts and immersion times of 10, 15, and 20 minutes, followed by sealing with acetic acid in a corrosive environment containing 3.5 % NaCl. The main goals were to evaluate the effectiveness of anodizing with and without sealing in lowering the rate of aluminum corrosion, to compare the effectiveness of anodizing with and without sealing, and to create adsorption models using Langmuir adsorption. Through the examination of the potentiodynamic approach, it was shown that anodizing had an inhibitory impact that was strengthened by sealing. The maximum efficiency of 76 % was attained after 20 minutes of anodizing and sealing at 10 volts. A correlation value of 0.7487 from the Langmuir adsorption modeling was also obtained, pointing to an advantageous adsorption behavior. This research demonstrates how effectively anodizing for aluminum alloy 2024 works with and without sealing, especially in a 3.5 % NaCl-corrosive environment

Keywords: Langmuir adsorption, aluminum alloy, acetic acid, inhibition efficiency, surface coverage

EFFECT OF ANODIZING ON ALUMINUM ALLOY 2024 WITH BORIC SULFATE ACID IN MEDIUM 3.5 % NaCl

Muhammad Zuchry

Doctoral Student, Graduate Student*

Ilyas Renreng

Corresponding Author

Doctorate, Professor*

E-mail: ilyas.renreng@gmail.com

Hairul Arsyad

Doctorate, Assistant Professor*

Lukmanul Hakim Arma

Doctorate, Assistant Professor*

*Department of Mechanical Engineering

Hasanuddin University

Malino str., Bontomarannu District, Gowa Regency,

South Sulawesi, Indonesia, 92171

Received date 25.05.2023

Accepted date 15.08.2023

Published date 30.08.2023

How to Cite: Zuchry, M., Renreng, I., Arsyad, H., Arma, L. H. (2023). Effect of anodizing on aluminum alloy 2024 with boric sulfate acid in medium 3.5 % NaCl. *Eastern-European Journal of Enterprise Technologies*, 4 (6 (124)), 41–50. doi: <https://doi.org/10.15587/1729-4061.2023.286351>

1. Introduction

Aluminum AA2024 is widely used in various industries due to its excellent strength-to-weight ratio and high fatigue resistance. However, one of its major drawbacks is its susceptibility to corrosion, especially in corrosive environments such as saline solutions [1]. Corrosion can significantly compromise the structural integrity and performance of aluminum AA2024, leading to substantial economic losses and safety concerns. Researchers have explored various surface treatment techniques to mitigate the corrosion issue to enhance the material's resistance to corrosion. One promising approach is boric sulfate acid anodizing, which involves the electrochemical oxidation of the aluminum surface in a solution containing boric acid and sulfate ions [2].

Aluminum Alloy 2024 (AA2024) benefits significantly from surface treatments such as anodizing and sealing [3, 4]. The exceptional strength-to-weight ratio and strong fatigue resistance [4] of AA2024 make it a popular choice in various industries, including aerospace, automotive, and construction. However, corrosion may weaken AA2024's structure and shorten its lifespan. Submerging AA2024 in an electrolyte and applying a direct current is a standard method of anodizing the material. One may influence the resulting oxide layer's thickness and characteristics by manipulating process-specific variables including anodizing voltage, current density, immersion time, and temperature [5, 6]. The anodized finish displays

enhanced corrosion resistance and may be refined for further aesthetic or functional applications.

Corrosion is a multifaceted and persistent phenomenon that exposes metals' susceptibility to their environment's dynamic influences. The durability of metals is constantly tested by the relentless assaults orchestrated by the elements, resulting in a silent yet profound battle. The metal oxidation process initiates as metals react with the surrounding atmosphere, particularly with elements like oxygen, marking the beginning of a complex interaction. Water, a seemingly ordinary substance, plays a significant role in promoting chemical reactions and facilitating the movement of ions. This creates conditions that are highly conducive to corrosion [7].

Sealing is a subsequent step often performed after anodizing to enhance corrosion resistance and improve the durability of the anodized surface. Sealing involves the closure of the pores present in the anodized oxide layer, making it more resistant to penetration by corrosive agents. Common sealing methods include hot water sealing, chromate conversion coating, or proprietary sealing solutions [8, 9]. The choice of sealing method depends on the specific requirements of the application and the desired properties of the sealed anodized surface. Combining anodizing and sealing processes significantly improves the corrosion resistance, surface hardness, wear resistance, and overall durability of AA2024. The anodized and sealed surface provides extended protection

against corrosive environments, ensuring the longevity and reliability of the aluminum alloy.

Boric sulfate acid anodizing has drawn interest as a potential strategy for slowing the rate of corrosion of aluminum AA2024. This procedure creates a shield between the alloy's surface and the corrosive environment in the form of a protective oxide layer. The material's mechanical characteristics and corrosion resistance are both improved by the anodized coating. There are various benefits to using boric sulfate acid as an anodizing electrolyte [10]. Boric acid provides buffering properties, maintaining a stable pH during the anodizing process. The presence of sulfate ions aids in the formation of a dense and adherent oxide layer. Additionally, this process can be performed at relatively low temperatures and with adjustable parameters, allowing for control over the resulting oxide layer's thickness and morphology [11, 12].

Boric sulfate acid's anodizing process may be modified to produce the required coating thicknesses and surface properties by modifying parameters such as voltage, current density, and anodizing duration [13, 14]. This allows for a greater degree of control over the final product. Because of this versatility, optimizing the anodizing process to meet specific performance criteria and achieve the required results is possible. In both scientific and commercial contexts, the boric sulfate acid anodizing AA2024 approach has garnered much interest. Numerous researches have been conducted to study the effect that anodizing parameters, surface preparation processes, and post-treatment techniques have on the overall performance of AA2024 and its corrosion resistance. According to the results [14, 15], boric sulfate acid anodizing is a successful method for lowering the corrosion rate, improving surface characteristics, and extending the amount of time AA2024 may remain in operation in settings where corrosion is present.

Current research is being conducted to explore the application of the anodizing method for surface coating in various studies. One of the objectives is to possess resistance against corrosion. Aluminum is a highly utilized material in multiple industries. The aluminum material tends to rust quickly. Therefore, it is crucial to implement a suitable coating method to protect it. Anodizing is commonly employed for applying a protective coating to aluminum materials. Different solutions are used to identify the most appropriate coating solution for aluminum. Therefore, studies devoted to developing anodizing methods for coating aluminum are relevant.

2. Literature review and problem statement

Numerous investigations have examined various methodologies for surface treatment aimed at mitigating the corrosion rate of Aluminum Alloy 2024 (AA2024) owing to its inherent vulnerability to corrosion. One approach that has been receiving increasing interest is Boric Sulfate Acid Anodizing. This process includes the deliberate electrochemical oxidation of the aluminum surface in an electrolyte solution that contains boric acid and sulfate ions [13, 16].

The paper [17] focuses on an innovative approach to anodization, utilizing organic additives in the sulfuric acid electrolyte. The quality of the anodized surface is assessed by many characterizations presented in the paper, including scanning electron microscopy (SEM), hardness tests, and thickness measurements. The results are more likely to be accurate because of this all-encompassing method. However,

this study only used a small sample size, and data on how easily these results may be replicated is lacking. The reliability of the research might be improved with a bigger sample size and by regularly repeating the studies. The process's potential uses are limited because sulfuric acid electrolyte with organic additions is required. Increasing the anodization method's adaptability would need investigating its compatibility with alternative electrolytes or process changes.

The study conducted in the paper [13] examines the influence of the sealing process on the corrosion resistance of AA2024 aluminum alloy. The research utilizes a suitable methodology involving anodizing with boric sulfuric acid, sealing the specimens, and performing corrosion resistance tests. The study uses corrosion resistance tests, and precise weight loss measurements, to objectively evaluate the efficacy of the sealing process. The purpose of these tests is to gather quantitative data that can be used to evaluate the effectiveness of the corrosion protection provided by the anodized aluminum. The article does not thoroughly analyze the underlying mechanisms involved in the sealing process and its impact on improving the corrosion resistance of anodized aluminum. The report lacks information regarding the sample size utilized in the study, which hinders the ability to assess the statistical significance of the findings. Additionally, there is limited data on the variability of the results, making it difficult to determine the reliability and generalizability of the study's conclusions. The article lacks a comparison of the corrosion resistance of the sealing process with other commonly used corrosion protection techniques, such as different sealing agents or alternative surface treatments.

The research [18] investigated the anti-corrosion capabilities of organic-based sealants on anodized AA2024T3 metal. Organic-based sealing is compared with more traditional techniques of corrosion prevention. The potential of organic-based species as a replacement corrosion prevention method may be gauged by comparing their performance to conventional methods. There is a lack of statistical analysis to determine the significance of the data, and the article does not give information regarding the sample size utilized in the research. The validity and applicability of the results might improve with a bigger sample size and proper statistical testing. To what extent employing organic species for sealing affects the environment or is sustainable is not investigated. The study's significance would be increased if it included the environmental effects of the sealing procedure.

The paper [6] examines using a sulfuric acid-free solution for hard anodizing, which offers a distinct and unconventional alternative to the commonly used sulfuric acid-based anodizing method. The potential outcome of this could be the identification and development of novel corrosion protection methods that are both effective and environmentally sustainable. The article compares the corrosion resistance between AA2024 and hard anodizing in a sulfuric acid-free solution, as opposed to the more commonly used sulfuric acid-based anodizing method. The comparative analysis offers valuable insights into the potential benefits and drawbacks of the innovative approach. The article's exclusive focus on aluminum alloy AA2024 may restrict the applicability of the results to other aluminum alloys or commonly used metal materials in different industries. The report lacks statistical analysis of the experimental data, including error bars or confidence intervals. Statistical analysis plays a critical role in assessing the significance of research outcomes and bolstering the reliability and trustworthiness of the findings.

The article [19] comprehensively analyzes the current literature on anodizing aerospace aluminum alloys for corrosion protection. The review offers an in-depth analysis of aerospace aluminum alloys, focusing on the impact of anodizing techniques on different materials frequently employed in the aerospace sector. The review primarily examines the effectiveness of anodized aerospace aluminum alloys in preventing corrosion. The review lacks a direct comparison of the efficacy of various anodizing methods and fails to analyze their respective advantages and disadvantages thoroughly. A comparative analysis can provide readers with a better understanding of the most suitable techniques for specific aerospace applications. The article lacks an in-depth analysis of the environmental consequences of different anodizing techniques.

Despite the shown effectiveness of boric sulfate acid anodizing in reducing the susceptibility of Aluminum AA2024 to corrosion, there are still unresolved issues that need remediation. The existing body of research primarily investigates the impact of anodizing and the subsequent buildup of the oxide layer on the corrosion resistance properties. However, more investigation is required to ascertain the durability and longevity of anodized coatings in varying conditions and over extended periods. Further research is needed to determine the influence of other variables, including surface preparation methodologies, post-treatment procedures, and alloying elements' impact on the corrosion behavior of boric sulfate acid anodized AA2024. In order to optimize the efficacy of the anodizing process and ensure the long-term durability of the rust-resistant properties conferred by anodized coatings, it is necessary to possess a comprehensive understanding of the following factors.

All this allows to assert that it is expedient to conduct a study on anodizing AA2024 using boric acid sulfate in 3.5 % sodium chloride. Anodizing is done to aluminum in order to strengthen its resistance to corrosion, which is one of the aims of the process. A thin coating of oxide can be produced by the anodizing process and applied to the surface of the material. When applied to aluminum material, the anodizing process, as opposed to the coating process, is the more suited option. Finding the appropriate solution to apply throughout the anodizing process and the sealing step might be difficult because of this. All of this provides us with the opportunity to underline how important it is to undertake research to evaluate the effect of anodizing using boric acid sulfate on AA2024 when it is immersed in 3.5 % NaCl medium.

3. The aim and objectives of the study

The aim of the study is to identifying the influence of boric sulfuric acid anodizing (BSAA) in a 3.5 % NaCl environment on corrosion resistance of AA2024.

To achieve this aim, the following objectives are accomplished:

- to investigate the impact of boric sulfate acid anodizing with sealing on the corrosion rate of AA2024 in a 3.5 NaCl environment;
- to examine the influence of boric sulfate acid anodizing with sealing on the inhibition efficiency of AA2024 in a 3.5 NaCl environment;
- to investigate the impact of boric sulfate acid anodizing with sealing on the anodizing thickness and conduct SEM analysis of AA2024 in a 3.5 % NaCl environment.

4. Materials and methods of research

4. 1. Object and hypothesis of the study

The object of this study is corrosion resistance of AA2024 aluminum alloy inside a corrosive environment, including a 3.5 % sodium chloride (NaCl) solution. The subobject of this study the effects of anodization, especially using boric sulfate acid, on the corrosion resistance of alloys when subjected to a salty environment. This study examines the correlation between several anodization factors, such as voltage, anodizing time, the sealing technique, and the resultant corrosion resistance of the alloy. Furthermore, the study aims to employ scanning electron microscopy (SEM) to investigate the surface features and properties of the anodized surfaces. This will contribute to a deeper comprehension of changes in surface structure and the creation of oxide layers.

The study's primary hypothesis is that boric sulfate acid anodization increases aluminum alloy 2024's corrosion resistance in a 3.5 % sodium chloride environment. The idea states that anodization forms an oxide coating on the alloy's surface, minimizing corrosion and improving seawater resilience. Boric sulfate acid anodization creates a stable and effective oxide coating on aluminum alloy. This oxide layer will protect the material against NaCl corrosion. A sealing technique following anodization improves alloy corrosion resistance. The sealing procedure is anticipated to plug oxide layer pores and fissures, increasing its density and impermeability and preventing corrosion. The hypothesis predicts that Langmuir adsorption modeling will show a positive association between corrosion inhibition and anodized surface adsorption. A more significant correlation coefficient indicates a stronger association between the anodized layer's corrosion-inhibiting characteristics.

The study may have relied on numerous assumptions to inform the research methodology and the interpretation of findings. The analysis assumes that the aluminum alloy 2024 employed in the trials had a uniform and homogenous composition devoid of notable fluctuations in alloying components that might potentially impact the anodization process or corrosion characteristics. The research may assume that the corrosion conditions in the 3.5 % NaCl medium are in a state of equilibrium, hence enabling accurate and consistent measurements of corrosion rates within the designated periods. The study posits that the sealing procedure has the potential to efficiently occlude the pores and fissures present in the oxide layer, hence augmenting its protective characteristics without inducing any harmful consequences. The study may posit that extraneous variables or impurities that have the potential to influence the anodization process or corrosion characteristics are mitigated or regulated throughout the experimental procedures.

The research study may have employed various simplifications to streamline the experimental procedures and improve data processing. The investigation might simplify the alloy composition analysis by exclusively considering AA2024, disregarding any composition discrepancies across various alloy batches. The research may assume idealized corrosion conditions in a 3.5 % NaCl media without considering potential differences in the composition, temperature, and other parameters in real-world situations. The study has the potential to streamline the investigation of corrosion behavior by specifically examining the isolated impacts of anodizing and sealing while disregarding potential interactions with other environmental variables.

4. 2. Material

The 12 mm in diameter, 3 mm thick aluminum alloy AA2024 was the primary focus of the investigation. The anodizing procedure was carried out at a controlled temperature of between 27 and 29 degrees Celsius and a steady voltage of 10 V. The anodizing electrolyte was a solution of 45 gr/l sulfuric acid and 8 gr/l boric acid, known as boric sulfuric acid anodizing (BSAA).

The anode and cathode were kept at a constant distance of 5 cm apart during the anodizing procedure. Both 10 and 15 minutes were allotted for the anodizing process. The roughness of the surface and the rate of corrosion after anodizing were two of the primary mechanical properties studied. The anodized layer's protective qualities were then improved by an additional sealing procedure. 0.5 % silicon and iron, 3.9 % copper, 0.6 % manganese, 1.5 % magnesium and titanium, 0.25 % zinc, 0.1 % chromium, and the rest 92.5 % was aluminum (in wt %) made up the AA2024 aluminum alloy utilized in the research.

To establish the basic metal's mechanical strength, its tensile strength was also assessed. The highest tensile stress (T_{max}) measured during the tensile test was 463 MPa, the yield stress (Y_{max}) was 360 MPa, and the elongation (E) measured at 17.8 %. These values fell within the acceptable ranges outlined in AA2024.

4. 3. Anodizing and sealing process

Aluminum alloy AA2024 with a diameter of 12 mm and a thickness of 3 mm was used in the testing setup. At a temperature of 27 °C, anodizing was carried out at a constant voltage of 10 V for periods of 10, 15, and 20 minutes. The BSAA electrolyte, which was composed of a combination of 45 gr/l sulfuric acid and 8 gr/l boric acid, served as the experiment's primary controlled variable. The cathode and anode were placed five centimeters apart from one another. The electrolyte mixture ratio for aluminum anodizing typically falls within the range of 30.5 to 52.0 g/l sulfuric acid and 5.2 to 10.7 g/l boric acid. However, the focus of this investigation was on employing a 45 g/l sulfuric acid/eight g/l combination [6].

The repair sequence before anodizing involved several steps. It began with degreasing and cleaning using a 10 gr/l sodium hydroxide (NaOH) solution, followed by rinsing with reverse osmosis (RO) water. Subsequently, etching was carried out using a caustic soda solution with a concentration of 100 gr/l, followed by rinsing. After that, desmutting was carried out for two minutes at a temperature between 27 and 32 degrees Celsius using a solution made up of 75 % phosphoric acid (H_3PO_4), 15 % sulfuric acid (H_2SO_4), and 10 % acetic acid (CH_3COOH). After washing, an electrolyte solution containing 45 gr/l sulfuric acid and 8 gr/l boric acid was used to carry out the anodizing procedure. The sealing process was then completed using a 50 gr/l solution of acetic acid (CH_3COOH). Fig. 1 illustrates the repair procedure and following processes of anodizing and sealing as part of the preparatory plan for the anodizing process.

The anodizing apparatus's schematic layout is seen in Fig. 2. Two samples were immersed in an electrolyte bath of sulfuric acid and boric acid to complete the anodizing

process. Both sulfuric acid (45 gr/l) and boric acid (8 gr/l) were used in the electrolyte. The anodizing process was carried out at a constant 10 volts for 10, 15, and 20 minutes. In this configuration, one specimen (made of the aluminum alloy AA2024) was used as the anode (+) and the other as the cathode (-). The voltage source for the anodizing process was established by connecting the cathode to the power supply.

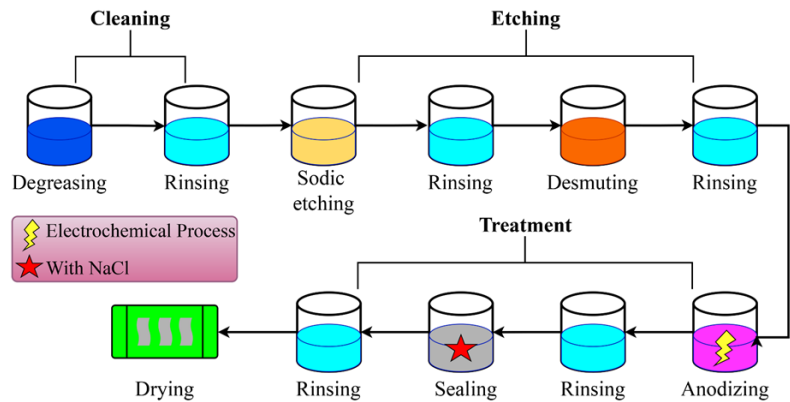


Fig. 1. Preparation scheme for anodizing process

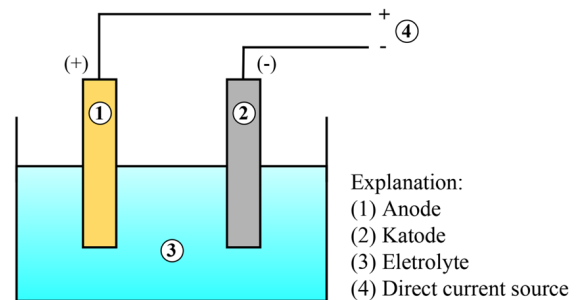


Fig. 2. Diagrammatic representation of the anodizing apparatus [1]

4. 4. Weight loss measurement

The experiment on weight loss adhered to the approach that was used, in which aluminum coupons were made and totally suspended in 1.4 M HNO_3 solutions, either with or without varying doses of Anisaldehyde. Glass hooks were utilized to suspend the coupons at a temperature of 308 K for 3 hours. The solution volume was maintained at 100 cm^3 . After the 3-hour immersion period, the coupons were retrieved, rinsed with distilled water, thoroughly dried, and reweighed. The corrosion rate in mg/cm^2 was then calculated based on the weight loss data obtained. The weight loss and corrosion rate of aluminum in the 1.4 M HNO_3 solution were determined for the solution without Anisaldehyde and the solutions with concentrations of 0.02, 0.04, 0.06, 0.08, and 0.10 M of Anisaldehyde. These calculations were performed using the provided equation, utilizing the weight loss data obtained. (1) shows the estimate of weight loss during the anodization process [19]:

$$\text{Weight loss } (\Delta W) = W_o - W_i \tag{1}$$

The surface coverage (Θ) and inhibition efficiency (I.E.) of varying inhibitor concentrations in acidic media were determined through weight loss experiments. Surface coverage (Θ) is calculated using the (2) [19]:

$$\text{Surface coverage} = (W_o - W_i) / W_o \tag{2}$$

The inhibition efficiency (I.E.) is calculated using the (3) [19]:

$$\text{Inhibition efficiency} = ((W_o - W_i) / W_o) \times 100 \%, \quad (3)$$

where W_o represents the initial weight of the specimen before immersion, and W_i represents the weight of the specimen after immersion in the acidic media.

The inhibition efficiency is expressed as a percentage and represents the inhibitor's effectiveness in reducing the specimen's weight loss. These equations provide a quantitative assessment of the degree of surface coverage and the inhibitor's effectiveness in protecting the specimen against corrosion in acidic environments.

4. 5. Scanning electron microscopic analysis

The surface of the aluminum sample was analyzed using a scanning electron microscope (SEM), specifically the FEI In-spect F50, to determine the thickness of the oxide layer formed after the anodization process. The SEM analysis was also conducted on aluminum samples that had undergone the sealing process. The SEM test was performed at a magnification of 20 μm , allowing for detailed examination and measurement of the oxide layer thickness. This characterization technique provides valuable insights into the structural and morphological properties of the anodized aluminum surfaces, aiding in evaluating the effectiveness of the anodization and sealing processes.

5. Results of the experiment using AA2024 anodized in boric sulfuric acid in a 3.5 % NaCl environment

5. 1. Corrosion rate on AA2024 in 3.5 % NaCl environment

Using the Boric Acid and Sulfuric Acid (BASA) approach, an essential quantitative examination of AA2024 was the measurement of the corrosion rate. The corrosion rate of diverse samples, including raw, unsealed, and sealed materials, is evaluated by this measurement. Additionally, the length of the anodizing procedure affects how quickly the material corrodes. The corrosion rate seen at a 10-volt anodizing voltage is shown in Fig. 3. This graphic gives a visual depiction of the data on corrosion rates and provides insightful information about how anodizing settings affect the corrosion resistance of the material.

Based on the obtained results, it is evident that incorporating a sealing process following anodization significantly reduces the corrosion rate across different anodizing time variations. The average corrosion rates for each variation were below 0.6 mmpy, indicating improved corrosion resistance. The corrosion rate actually lowered by up to 15 % after applying the sealing technique for a 10-minute anodizing period. Similar to this, after sealing, the corrosion rates for anodizing times of 15 and 20 minutes fell by 11 % and 0.2 %, respectively.

It is also interesting that, regardless of whether the samples were sealed or left unsealed, the direct anodization period alone helped to lower the corrosion rate. Unsealed samples saw a 47 % reduction in corrosion rate from their initial state. The corrosion rate was reduced by 45 % in the unsealed samples, compared to 45 % in the sealed samples. These results show how the sealing procedure and the anodization period work together to reduce corrosion and increase the durability of the aluminum samples.

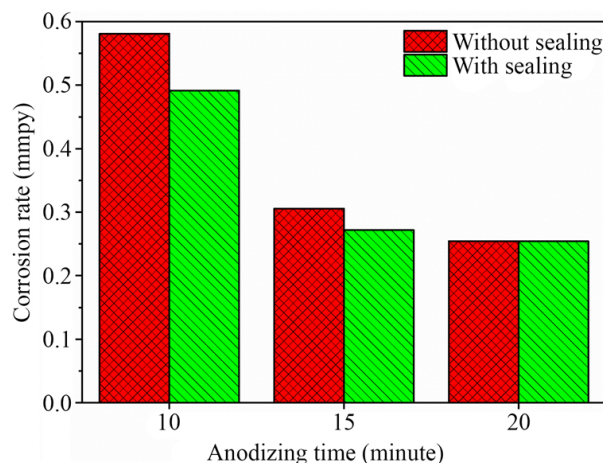


Fig. 3. Corrosion rate on anodized specimens

5. 2. Inhibition efficiency on AA2024 in 3.5 % NaCl environment

The inhibition efficiency of AA2024 in a 3.5 % NaCl environment was evaluated, and the results showed significant corrosion protection. The inhibition efficiency, calculated based on weight loss measurements and other corrosion-related parameters, indicated that the surface treatment, most likely anodizing, effectively reduced the corrosion rate of AA2024 in the aggressive NaCl environment. The inhibition efficiency values obtained demonstrated that the treated AA2024 specimens exhibited improved resistance against corrosion when exposed to the 3.5 % NaCl solution compared to untreated samples (Fig. 4).

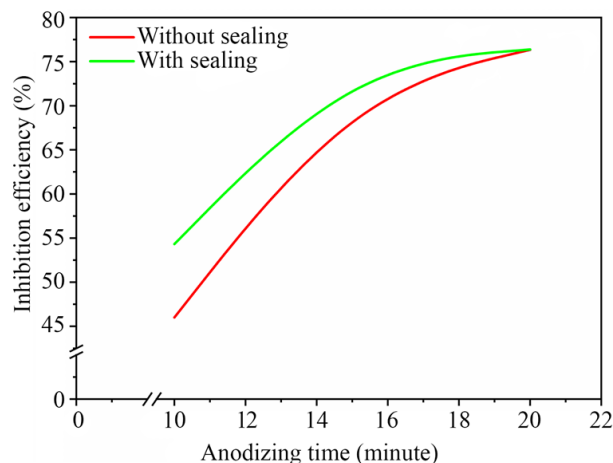


Fig. 4. The inhibition efficiency observed in anodized specimens

When examining the inhibition efficiency, a noticeable trend emerges, indicating that the efficiency increases after the sealing process is applied to the specimens. Moreover, as the anodizing time is extended, the efficiency demonstrates a positive correlation, showing a progressive improvement. The results indicate that up to a 20-minute anodizing time, the inhibition efficiency of specimens with sealing is nearly equivalent to that of samples without sealing.

The similarity in inhibition efficiency between sealed and unsealed specimens for up to 20 minutes may suggest that the anodizing process provides significant corrosion

protection. However, it is essential to note that the sealing process still contributes to enhancing the overall effectiveness of the surface treatment technique, providing additional durability and extended service life, especially in more challenging corrosive environments.

The surface coverage results on AA2024 in the 3.5% NaCl environment demonstrate a significant improvement after the sealing process illustrated in Fig. 5. The data indicates that the specimens' surface coverage increases following the sealing step's application. As the anodizing time is extended, the surface coverage also significantly increases. Up to 20 minutes of anodizing time, the surface coverage of specimens without sealing and those with sealing show a similar trend, with both achieving substantial coverage.

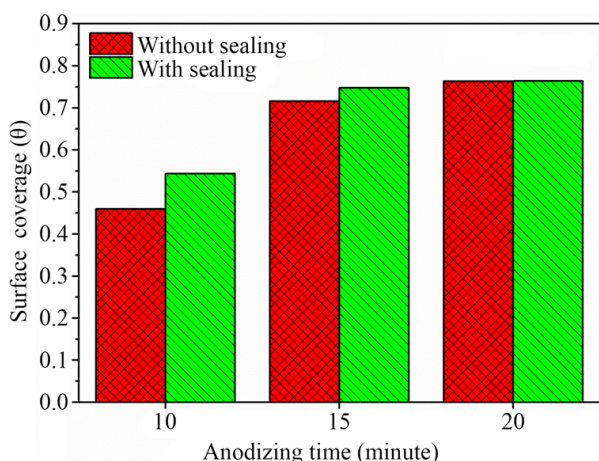


Fig. 5. The extent of surface coverage observed in anodized specimens

The surface coverage percentage rose when comparing the samples without sealing to those with the sealing process at anodizing durations of 10 minutes, 15 minutes, and 20 minutes, with increases of 18%, 4%, and 0.05%, respectively. These findings suggest that the length of the anodizing time impacts the surface coverage of aluminum. Moreover, implementing a sealing process after anodizing can further enhance the surface coverage on the aluminum surface layer. In summary, both the anodizing time and the sealing process play vital roles in improving the aluminum material's surface coverage and protective properties.

Fig. 6 presents the outcomes of the Langmuir adsorption modeling, illustrating a comparison between specimens without and those with sealing. Since the adsorption process is chemical, it has been appropriately modeled using the Langmuir adsorption method. This modeling approach provides a comprehensive understanding of the adsorption behavior and valuable insights into the interaction between the inhibitors and the aluminum surface during the sealing process. By utilizing Langmuir adsorption modeling, researchers gain valuable information about the adsorption capacity and affinity of the inhibitors, further contributing to the understanding of the corrosion protection mechanisms and the effectiveness of the sealing process in enhancing the surface properties of the aluminum alloy.

The R2 values for each material under different types of isothermal adsorption conditions were obtained by analyzing the two curves presented above. The R2 value represents the coefficient of determination, providing insights into the influence of the independent variable (X) on the dependent

variable (Y). Ranging from 0 to 1, a value closer to 1 indicates a stronger influence of the independent variable on the dependent variable and vice versa. In this case, the correlation coefficient results for specimens without seals and specimens with seals were found to be 0.6526 and 0.7487, respectively.

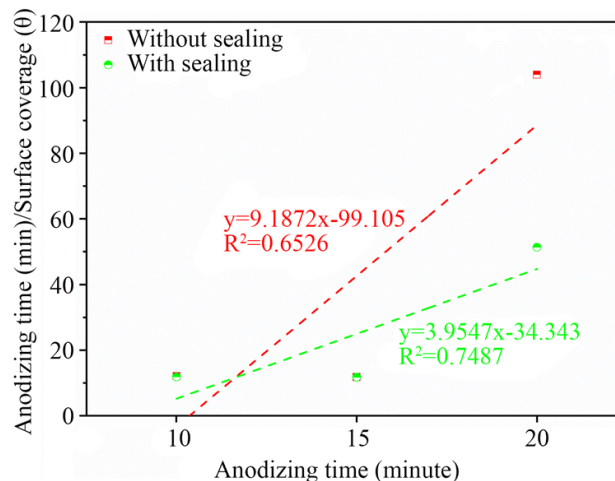


Fig. 6. The results of Langmuir adsorption modeling

The analysis of electrochemical polarization provides vital insights into the system's behavior, and these discoveries are well depicted by the Tafel curve plot illustrated in Fig. 7. The described plot functions as a graphical illustration of the correlation between current density and electrode potential. Our study focuses on two unique groups of specimens subjected to an anodizing voltage of 10 volts: one group undergoes the anodizing procedure without subsequent sealing, while the other group is sealed after anodization. The implementation of distinct treatment methods enables us to investigate the influence of sealing on the electrochemical properties of the specimens.

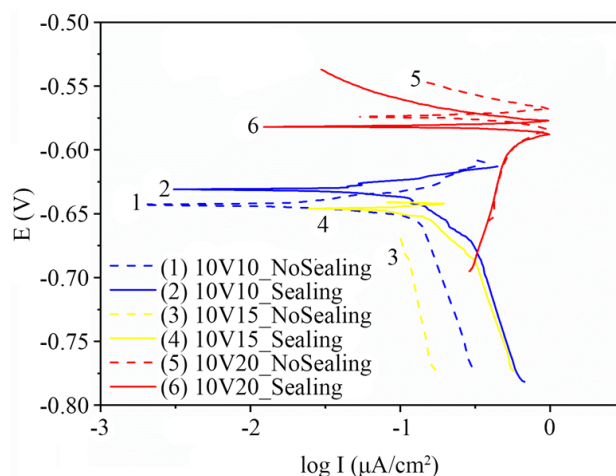


Fig. 7. The polarization curves obtained at an anodizing voltage of 10 volts

The sealed specimens demonstrate a significant increase in potential compared to the non-sealed counterparts. The observed divergence in the electrochemical behavior of the samples indicates that the sealing process has a noticeable impact on their performance. Furthermore, it is crucial

to emphasize that the corrosion potential increases as the duration of anodizing is extended. The observed correlation highlights the complex connection between the time of anodization and the changes in corrosion properties. The Tafel curve plot is a valuable tool for analyzing the relationship between anodization parameters and the electrochemical responses of aluminum alloy specimens. It visually represents these trends, providing a clear understanding of how different parameters affect the overall behavior of the samples. The visual representation enhances our comprehension of the corrosion protection mechanisms facilitated by anodization and sealing processes.

5. 3. Thickness of anodizing and scanning electron microscopic analysis on AA2024 in 3.5 % NaCl environment

Both specimens with and without sealing were used in the study of the thickness of the oxide layer after anodization, as shown in Fig. 8. On the aluminum's surface, the oxide layer's thickness was measured in five different places. It is crucial to remember that varied anodizing durations result in differing oxide layer thicknesses, which have a big impact on how resistant to corrosion aluminum is.

The oxide layer's thickness is a crucial determinant of the aluminum alloy's corrosion protection capabilities. A thicker oxide layer generally enhances the material's resistance to corrosion, as it acts as a more robust barrier against the detrimental effects of the surrounding environment. Consequently, anodizing processes with longer durations are expected to yield thicker oxide layers, thereby improving corrosion resistance for the aluminum material.

However, the presence of a sealing procedure following anodization might also affect how thick the oxide layer is. As a result of the sealing procedure, the oxide layer's holes and fissures are sealed off, increasing its density and boosting its protective qualities. As a result, the oxide layer on the surface of the aluminum becomes thicker and more impermeable, increasing its ability to resist corrosion. The average thickness of the oxide layer produced by the anodizing process is shown in Fig. 9. The results show that specimens without sealing have an oxide layer that is thicker than specimens with sealing. The length of the anodizing process also has a considerable impact on how thick the oxide layer is. The oxide layer thickens with longer anodizing times.

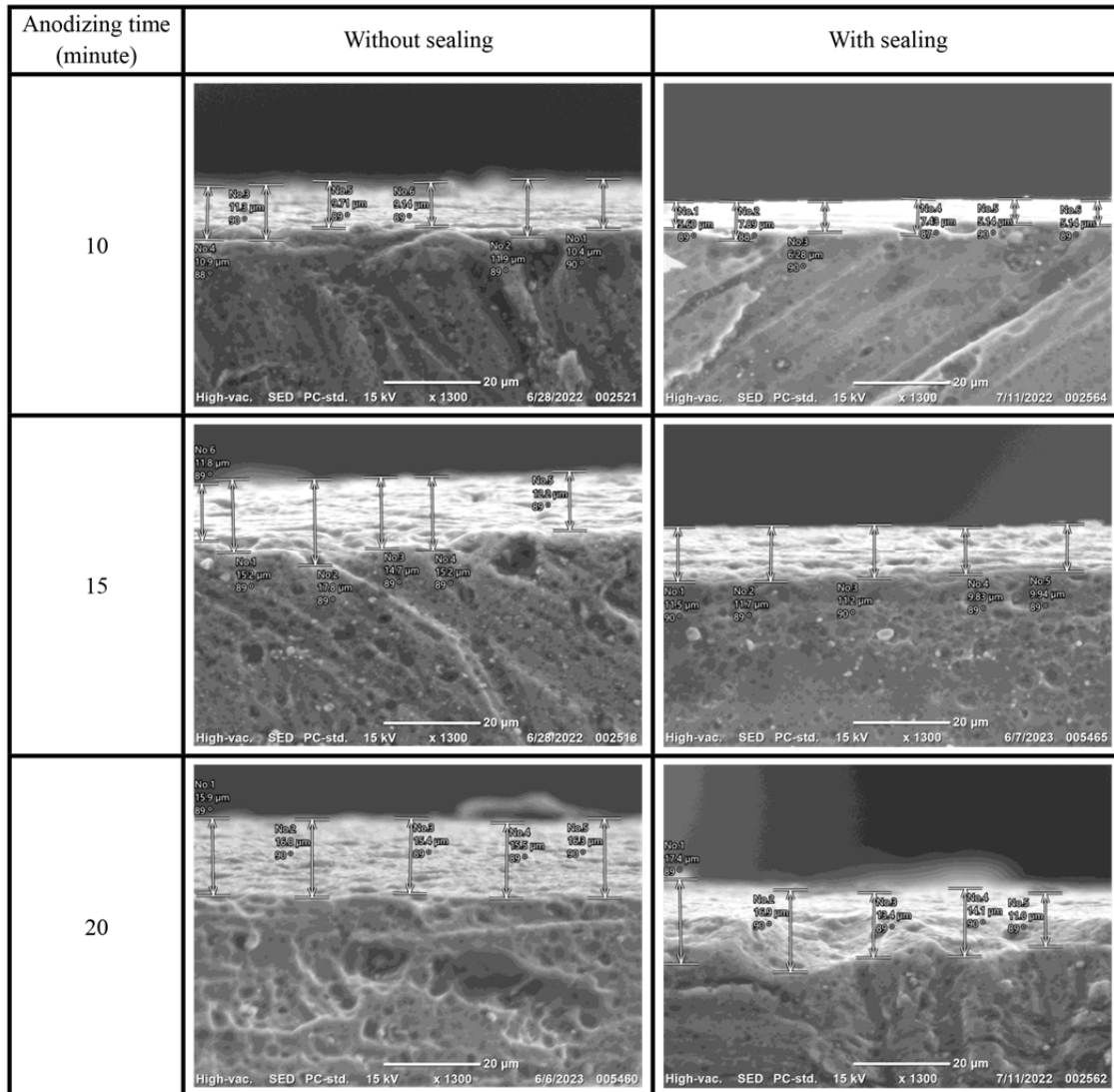


Fig. 8. The scanning electron microscopic image of the aluminum surface following the anodizing process

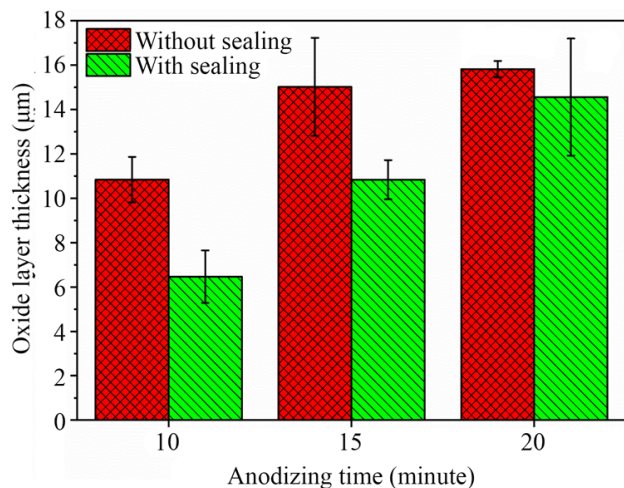


Fig. 9. The average thickness of the oxide layer for each anodization parameter

The thickness of the oxide layer observed in both specimens remains below 16 μm , indicating that the anodization process does not lead to excessively thick coatings. Instead, the oxide layer formed is within a controlled and desirable range for optimal corrosion protection. For specimens without the sealing process, the oxide layer experienced an increase of up to 46 % when subjected to an anodizing time of 20 minutes. On the other hand, specimens with the sealing process exhibited a more substantial increase of up to 125 % in the oxide layer thickness at the same anodizing time of 20 minutes.

6. Discussion of the experiment that involved anodizing AA2024 in a 3.5 % NaCl environment using boric sulfuric acid

The corrosion rates measured for different anodizing durations were consistently below 0.6 mmpy on average. This suggests that the specimens with varying anodizing times exhibited a greater resistance to corrosion compared to the unsealed specimens, as shown in Fig. 3. The study demonstrated that longer periods of anodizing result in more significant reductions in corrosion rates. This suggests that the sealing process becomes increasingly important as the duration of anodizing increases. The primary finding of this study indicates that the incorporation of a sealing process following anodization leads to a significant decrease in the corrosion rate observed in aluminum samples. The sealing method is an effective way to protect aluminum from corrosion. It creates a barrier that prevents direct contact between the aluminum and corrosive substances in the environment [8]. The investigation highlights that the effectiveness of the sealing procedure is influenced by the timing of anodization [13]. The correlation between the duration of anodizing and the subsequent decrease in corrosion rate becomes more evident as the duration of anodizing is increased, especially after the sealing procedure is applied. The discovery highlights the importance of modifying the duration of anodization in order to achieve the desired level of corrosion resistance that is suitable for specific applications.

The sealing procedure was successful in closing all of the holes that were present inside the anodized oxide layer,

which led to an increase in the inhibition efficiency once sealing was completed. As a direct result of this, the protective properties of the coating have been improved, which has led to an increase in its resistance to corrosive substances. As a result, sealing is of the highest significance to give greater corrosion resistance to the treated specimens, particularly in situations where the anodizing treatments are carried out over a longer period of time [20].

Fig. 6 displays the estimated values of the correlation coefficient for samples sealed and unsealed in this study. The results of this investigation support the hypothesis that isothermal adsorption is favorably affected by the post-anodization sealing procedure. A stronger association between the independent and dependent variables is shown by the higher correlation coefficient of 0.7487 found in the sealed samples compared to the unsealed specimens' value of 0.6526 (Fig. 6). The results show that the efficacy of isothermal adsorption is much improved by the sealing method [21]. According to the data, the adsorption and interaction of the adsorbate molecules with the aluminum surface are enhanced by the sealing operation, as evidenced by the higher R^2 value reported for sealed specimens [22]. These findings highlight the potential benefits of employing sealed samples in real-world circumstances requiring increased resistance to corrosion, and also highlight the significance of the sealing method in boosting the overall adsorption capabilities.

The correlation coefficient value of 0.7487 obtained for specimens with seals indicates a strong relationship between the variables. This suggests that the sealing procedure significantly affects the isothermal adsorption behavior, as shown in Fig. 6. The statement suggests that the purpose of sealing is to improve the interaction between inhibitors and the aluminum surface. This enhancement is believed to enhance the adsorption properties and offer better protection against corrosion. The correlation coefficient indicates a strong positive relationship between the post-anodizing procedure and the corrosion resistance of the aluminum alloy. This suggests that implementing this procedure can be highly beneficial in improving the alloy's resistance to corrosion. The coefficient suggests that the sealing approach leads to a more advantageous and effective adsorption process [23].

The results highlight the sealing process's significant impact on the oxide layer's thickness. Applying a sealing step after anodizing increases the oxide layer's density significantly, resulting in a thicker and more effective barrier against corrosion. This phenomenon is especially prominent in specimens subjected to longer anodizing times, as the sealing process contributes to greater oxide layer growth [24]. The information obtained from these observations is valuable for understanding the relationship between anodizing conditions, sealing processes, and the resulting oxide layer characteristics. Such insights can aid in optimizing the surface treatment technique to achieve the desired corrosion protection performance for aluminum materials in various industrial applications [25].

This study has limitations, including its narrow emphasis on a single corrosive media (3.5 % NaCl). The results may not indicate the aluminum alloy's performance in other corrosive conditions, but they give valuable insights into its corrosion resistance. Corrosion processes and behaviors might change depending on the environment's aggressiveness and chemical composition. Mass loss experiments as a proxy for corrosion rate and inhibition efficiency also have limitations. Despite their popularity and the helpful information they

give, trials designed to induce weight reduction have several apparent drawbacks. Corrosion can't be monitored using weight loss studies in real time since variables like exposure duration, surface condition, and handling practices might affect the findings. The importance of localized corrosion events in certain real-world contexts may also be missed in weight-loss research.

Further investigation can be conducted to enhance the development of this research. There are several aspects that may be further explored. By utilizing sophisticated microstructural analysis techniques, such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), and X-ray diffraction (XRD), it becomes possible to discern the alterations in the oxide layer's structure and gain a deeper understanding of the interface between the anodic film and the substrate. Furthermore, it is important to conduct comparative analyses using alternative corrosion protection methods, such as coatings, inhibitors, and composite materials, in order to assess the relative efficacy and constraints of anodization in various scenarios. The application of computer modeling and simulation approaches is employed to forecast and comprehend the corrosion behavior and creation of oxide layers under diverse situations, hence facilitating the development of more efficient anodization procedures.

7. Conclusions

1. Sealing significantly reduces corrosion across anodizing times. Average corrosion rates were below 0.6 mmpy, indicating improved resistance. Sealing reduced corrosion by 15 % at 10 minutes, 11 % at 15 minutes, and 0.2 % at 20 minutes. Direct anodization time also decreased corrosion, regardless of sealing. Unsealed samples saw a 47 % decrease, while sealed samples experienced a 45 % reduction. Both sealing and anodization time effectively enhance aluminum durability against corrosion.

2. Evaluating inhibition efficiency reveals a notable trend where sealing the specimens increases efficiency. With longer anodizing times, efficiency improves progres-

sively. Up to 20 minutes of anodizing, sealed samples show similar inhibition efficiency to unsealed ones. This suggests that anodizing alone offers significant corrosion protection. However, the sealing process further enhances the surface treatment, providing increased durability and extended service life, especially in harsh corrosive conditions.

3. The oxide layer thickness is a key component in influencing the aluminum alloy's resistance to corrosion. When the oxide layer protecting an object from the environment is made thicker, the object is better protected from corrosion. With a 20-minute sealing procedure and anodizing period, the average oxide layer thickness is below 16 microns, and there is an increase of up to 125 % on specimens.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

Financing

The study was performed without financial support.

Data availability

Data will be made available on reasonable request.

Acknowledgments

This research has been made possible with the valuable support and assistance from the Mechanical Engineering Laboratory at Hasanuddin University. The dedicated contributions and resources provided by the laboratory have contributed significantly to the successful execution and completion of this study.

References

- Merisalu, M., Aarik, L., Kozlova, J., Mändar, H., Tarre, A., Sammelselg, V. (2021). Effective corrosion protection of aluminum alloy AA2024-T3 with novel thin nanostructured oxide coating. *Surface and Coatings Technology*, 411, 126993. doi: <https://doi.org/10.1016/j.surfcoat.2021.126993>
- Zhang, J., Zhao, X., Zuo, Y., Xiong, J. (2008). The bonding strength and corrosion resistance of aluminum alloy by anodizing treatment in a phosphoric acid modified boric acid/sulfuric acid bath. *Surface and Coatings Technology*, 202 (14), 3149–3156. doi: <https://doi.org/10.1016/j.surfcoat.2007.10.041>
- Saeedikhani, M., Javidi, M., Yazdani, A. (2013). Anodizing of 2024-T3 aluminum alloy in sulfuric-boric-phosphoric acids and its corrosion behavior. *Transactions of Nonferrous Metals Society of China*, 23 (9), 2551–2559. doi: [https://doi.org/10.1016/s1003-6326\(13\)62767-3](https://doi.org/10.1016/s1003-6326(13)62767-3)
- Elabar, D., La Monica, G. R., Santamaria, M., Di Quarto, F., Skeldon, P., Thompson, G. E. (2017). Anodizing of aluminium and AA 2024-T3 alloy in chromic acid: Effects of sulphate on film growth. *Surface and Coatings Technology*, 309, 480–489. doi: <https://doi.org/10.1016/j.surfcoat.2016.11.108>
- Covelo, A., Rodil, S., Nóvoa, X. R., Hernández, M. (2022). Development and characterization of sealed anodizing as a corrosion protection for AA2024-T3 in saline media. *Materials Today Communications*, 31, 103468. doi: <https://doi.org/10.1016/j.mtcomm.2022.103468>
- Miramontes, J. C., Gaona Tiburcio, C., García Mata, E., Esneider Alcála, M. Á., Maldonado-Bandala, E., Lara-Banda, M. et al. (2022). Corrosion Resistance of Aluminum Alloy AA2024 with Hard Anodizing in Sulfuric Acid-Free Solution. *Materials*, 15 (18), 6401. doi: <https://doi.org/10.3390/ma15186401>

7. Schindelholz, E. J., Melia, M. A., Rodelas, J. M. (2021). Corrosion of Additively Manufactured Stainless Steels—Process, Structure, Performance: A Review. *Corrosion*, 77 (5), 484–503. doi: <https://doi.org/10.5006/3741>
8. Ofoegbu, S. U., Fernandes, F. A. O., Pereira, A. B. (2020). The Sealing Step in Aluminum Anodizing: A Focus on Sustainable Strategies for Enhancing Both Energy Efficiency and Corrosion Resistance. *Coatings*, 10 (3), 226. doi: <https://doi.org/10.3390/coatings10030226>
9. Wilcox, G. D., Walker, D. E. (2013). Chemical Conversion Coatings. *Encyclopedia of Tribology*, 359–366. doi: https://doi.org/10.1007/978-0-387-92897-5_1004
10. Zhang, L., Thompson, G. E., Curioni, M., Skeldon, P. (2013). Anodizing of Aluminum in Sulfuric Acid/Boric Acid Mixed Electrolyte. *Journal of The Electrochemical Society*, 160 (4), C179–C184. doi: <https://doi.org/10.1149/2.032306jes>
11. Chamidy, H. N., Ngatin, A., Rosyadi, A. F., Julviana, A., Noviyani, N. (2023). Effect of voltage on the thickness of oxide layer at aluminum alloys for structural bonding using phosphoric sulfuric acid anodizing (PSA) process. *International Journal of Mechanical Engineering Technologies and Applications*, 4 (1), 69–76. doi: <https://doi.org/10.21776/mechta.2023.004.01.8>
12. Raffin, F., Echouard, J., Volovitch, P. (2023). Influence of the Anodizing Time on the Microstructure and Immersion Stability of Tartaric-Sulfuric Acid Anodized Aluminum Alloys. *Metals*, 13 (5), 993. doi: <https://doi.org/10.3390/met13050993>
13. Zuchry, M., Renreng, I., Arsyad, H., Arma, L. H., Widianto, A. (2023). Sealing effect on corrosion resistance of boric sulfuric acid anodizing on AA2024. *Eastern-European Journal of Enterprise Technologies*, 2 (6 (122)), 43–52. doi: <https://doi.org/10.15587/1729-4061.2023.277961>
14. Naief, T. M., Rashid, K. H. (2011). Comparative Study for Anodizing Aluminum Alloy 1060 by Different Types of Electrolytes Solutions. *Engineering & Technology*. URL: https://www.researchgate.net/publication/295911313_Comparative_Study_for_Anodizing_Aluminum_Alloy_1060_by_Different_Types_of_Electrolytes_Solutions
15. Zarras, P., Stenger-Smith, J. D. (2015). Smart Inorganic and Organic Pretreatment Coatings for the Inhibition of Corrosion on Metals/Alloys. *Intelligent Coatings for Corrosion Control*, 59–91. doi: <https://doi.org/10.1016/b978-0-12-411467-8.00003-9>
16. Wan, Y., Wang, H., Zhang, Y., Wang, X., Li, Y. (2018). Study on Anodic Oxidation and Sealing of Aluminum Alloy. *International Journal of Electrochemical Science*, 13 (2), 2175–2185. doi: <https://doi.org/10.20964/2018.02.78>
17. Hou, F., Hu, B., Tay, S. L., Wang, Y., Xiong, C., Gao, W. (2017). A new, bright and hard aluminum surface produced by anodization. *International Journal of Modern Physics B*, 31 (16-19), 1744029. doi: <https://doi.org/10.1142/s0217979217440295>
18. Collazo, A., Ezpeleta, I., Figueroa, R., Nóvoa, X. R., Pérez, C. (2020). Corrosion protection properties of anodized AA2024T3 alloy sealing with organic-based species. *Progress in Organic Coatings*, 147, 105779. doi: <https://doi.org/10.1016/j.porgcoat.2020.105779>
19. Paz Martínez-Viademonte, M., Abrahami, S. T., Hack, T., Burchardt, M., Terryn, H. (2020). A Review on Anodizing of Aerospace Aluminum Alloys for Corrosion Protection. *Coatings*, 10 (11), 1106. doi: <https://doi.org/10.3390/coatings10111106>
20. Merino, E., Durán, A., Ceré, S., Castro, Y. (2022). Hybrid Epoxy-Alkyl Sol–Gel Coatings Reinforced with SiO₂ Nanoparticles for Corrosion Protection of Anodized AZ31B Mg Alloy. *Gels*, 8 (4), 242. doi: <https://doi.org/10.3390/gels8040242>
21. Aghaei, M., Anbia, M., Salehi, S. (2022). Measurements and modeling of CO₂ adsorption behaviors on granular zeolite 13X: Impact of temperature and time of calcination on granules properties in granulation process using organic binders. *Environmental Progress & Sustainable Energy*, 41 (5). doi: <https://doi.org/10.1002/ep.13866>
22. Hafeznezami, S., Zimmer-Faust, A. G., Dunne, A., Tran, T., Yang, C., Lam, J. R. et al. (2016). Adsorption and desorption of arsenate on sandy sediments from contaminated and uncontaminated saturated zones: Kinetic and equilibrium modeling. *Environmental Pollution*, 215, 290–301. doi: <https://doi.org/10.1016/j.envpol.2016.05.029>
23. Jedli, H., Jbara, A., Hedfi, H., Bouzgarrou, S., Slimi, K. (2017). Carbon dioxide adsorption isotherm study on various cap rocks in a batch reactor for CO₂ sequestration processes. *Applied Clay Science*, 136, 199–207. doi: <https://doi.org/10.1016/j.clay.2016.11.022>
24. Fedel, M., Franch, J., Rossi, S. (2021). Effect of thickness and sealing treatments on the corrosion protection properties of anodic oxide coatings on AA5005. *Surface and Coatings Technology*, 408, 126761. doi: <https://doi.org/10.1016/j.surfcoat.2020.126761>
25. Wang, R., Wang, L., He, C., Lu, M., Sun, L. (2019). Studies on the sealing processes of corrosion resistant coatings formed on 2024 aluminium alloy with tartaric-sulfuric anodizing. *Surface and Coatings Technology*, 360, 369–375. doi: <https://doi.org/10.1016/j.surfcoat.2018.12.092>