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# Overview of anodization and silver coating for titanium alloys: Process parameters and biomedical insights

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## Abstract

Anodization is a critical electrochemical process for producing titanium oxide layers with varying characteristics, significantly influencing the physicochemical properties, biocompatibility, and performance of bone implants. This study systematically reviews the current state of research on the effects of anodization parameters and silver coatings on the morphology, functional groups, and phase identification of Ti-6Al-4V bone implants. By synthesizing findings from 18 relevant studies selected from 1044 screened articles (2000–2023), this review provides a comprehensive framework for understanding the role of anodization and silver coating in improving implant performance. The review highlights how variations in anodization parameters—such as electrolyte composition, voltage, and duration-significantly impact critical implant properties, including corrosion resistance, antimicrobial efficacy, and biocompatibility. Additionally, silver coatings are underscored for their antimicrobial benefits and ability to address challenges such as bacterial adhesion and biofilm formation. Beyond functional improvements, this review identifies gaps in the literature, such as the limited exploration of process optimization and the environmental implications of implant fabrication, offering actionable insights for future research. The novelty of this article lies in its holistic synthesis of fragmented findings, bridging material science, biomedical functionality, and sustainability. It provides a structured evaluation of key process parameters and their influence on implant performance, emphasizing the need for balanced approaches that integrate clinical effectiveness with environmentally responsible practices. By offering a unified perspective, this review serves as a valuable reference for advancing both research and practical applications in the development of high-performance bone implants.

Keywords: Anodization; Ti-6Al-4V; Silver; Implant

# 1. Introduction

Anodization, an electrochemical process, has become a significant method of producing oxide layers of varying thickness on metal surfaces, with an example of its application to titanium metal producing TiO<sub>2</sub> films. Variations in electrolyte, time, and voltage in the anodization process can result in significant differences in the physicochemical characteristics, biocompatibility, mechanical strength, corrosion resistance, and even antimicrobial capabilities of bone implants. The process involves converting titanium metal into TiO2 polymers on the surface using an electrolyte medium such as ethylene glycol solution mixed with NH<sub>4</sub>F and water, where an electric current is delivered through an electrochemical system [1]. At the initial stage, the choice of electrolyte type is key

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Universitas Muhammadiyah Magelang because of its influence on the formation of an oxide layer that is not only thick but also stable, which significantly reduces the corrosion rate. Lee [2] found that the current flow and the formation of a thick barrier layer during anodization significantly affect the electrochemical characteristics and corrosion resistance. Furthermore, Ali [3] demonstrated that anodizing in sodium oxalate-sodium silicate solution can produce a stable anodic aluminum layer with tailored porosity and surface characteristics, leading to high corrosion resistance. Bestetti et al. [4] discussed the impact of electrolyte composition on the anodic oxidation process and oxide characteristics in arc-arc micro anodization of magnesium alloys. Correspondingly, Mahallawy [5] studied the anodization of AZ91 magnesium alloy using an environmentally friendly electrolyte, finding that anodic film thickness, surface morphology, microstructure, and corrosion resistance were affected by the electrolyte used. These studies together highlight the significant impact of electrolyte choice on the physicochemical characteristics, biocompatibility, mechanical strength, corrosion resistance, and biological response of anodized materials.

Furthermore, the anodization process, especially the duration or length of anodization time, significantly affects the morphological characteristics, phase groups, phase identification of the resulting material. Mehdizade [6] found that an increase in anodization time resulted in a decrease in wall thickness and an increase in pore diameter, which in turn increased the resistance to corrosion. Darmawan [7] also observed that a larger current during anodization resulted in a thicker oxide layer and increased corrosion resistance. Correspondingly, it was also found that a longer anodization duration led to the growth of a porous oxide layer containing sulfur, increasing the barrier ability of the material [8]. In addition, it was confirmed in another study that anodization time affects the corrosion resistance of the resulting film, with 60 min being optimal for good corrosion resistance [9]. Correspondingly, it was also found that time significantly affects not only the growth of the coating but also the characteristics of the protective film in a physiological environment [10]. Therefore, a deep understanding of these anodization parameters is important in achieving the desired uniformity and quality of oxide layers for various applications, including in the biomedical field. The last parameter, the gradually applied voltage plays an important role in determining the thickness of the oxide layer, as the formation of oxygen bubbles in the solution improves the homogeneity of the layer growth [11]. In addition, research by Setyarini [12] showed that stress also has a positive effect on the improvement of surface roughness and the reduction of pore size. Among the various surface modification methods available for titanium and its alloys-such as Physical Vapor Deposition (PVD), Plasma Spraying, Plasma Immersion Ion Implantation, Selective Laser Melting (SLM), Chemical Vapor Deposition (CVD), Sol-Gel, and Micro-Arc Oxidation (MAO)-anodization stands out for its simplicity, costeffectiveness, and ability to achieve precise control over oxide layer characteristics. Unlike other techniques, anodization enables the formation of highly uniform and adherent oxide layers with tunable thickness and morphology through adjustable electrochemical parameters. Additionally, the anodization process allows for straightforward integration with silver coating techniques, enhancing antimicrobial properties while maintaining biocompatibility. These advantages make anodization a practical and efficient choice for biomedical applications, particularly for bone implants where controlled surface modifications are critical for improving corrosion resistance, mechanical stability, and biological integration. For example, in the titanium alloy Ti-6Al-4V, anodization can improve corrosion resistance, but higher anodizing stresses can reduce these characteristics [13]. Similarly, the current density used in the anodization of AZ31B magnesium alloy can also affect surface chemistry, film morphology, and corrosion behavior, without compromising biocompatibility [14]. However, the mechanical characteristics of aluminum films anodized at high pressure may be negatively affected, with decreased fatigue life and increased crack initiation sites [15].

Titanium and its alloys have been the primary choice in surgical and bone replacement applications for the past 20 years due to their biocompatibility and corrosion [16]–[19]. Although Ti-6Al-4V is widely recognized for its biocompatibility and corrosion resistance, its inherent limitations, such as susceptibility to bacterial adhesion and biofilm formation, pose significant challenges in clinical applications [20]. These issues can compromise the longevity and effectiveness of implants, leading to post-operative complications such as infections. To address these drawbacks, surface modifications, particularly coatings like silver, have been extensively investigated. Silver coatings, known for their potent antimicrobial properties, provide an additional protective layer, mitigating infection risks and enhancing overall implant performance by preventing bacterial colonization and promoting tissue integration [21]. Furthermore, a study by Fowdar [21] mentioned 3 ways to reduce these drawbacks, namely by coating with zinc, copper,

and silver. Silver has numerous applications due to its antimicrobial action, and it is regarded as the finest inorganic antibacterial agent, capable of combating a wide range of infection-causing agents [21]. In current times, silver is employed in water treatment and the medical field. Silver's antibacterial characteristics are derived from its capacity to release silver ions (Ag+) when in contact with water. Ag+ is bioactive, meaning it can interact with proteins, amino acids, and receptors found in bacterial cell walls [22]. As a result, a higher ionization capacity will result in the release of more Ag+, and hence a greater antibacterial impact.

There are still very few systematic literature reviews that specifically examine the impact of anodizing process variations on titanium that is subsequently silver plated on the morphological characteristics, phase groups, and phase identification of bone implants, despite the fact that numerous studies have been conducted on the effect of anodizing process parameters based on electrolyte, time, and voltage on the surface characteristics of the resulting titanium material. In addition, it is important to review the literature on anodization process using silver plating method to see the extent of impact on physicochemical characteristics, biocompatibility, mechanical strength, corrosion resistance, and biological response in bone implants. With a focus on anodization parameters such as electrolyte, voltage, and time as well as silver plating methods. This study aims to fill this knowledge gap by summarizing recent and relevant findings in a systematic literature review. Specifically, the purpose of this study is to evaluate the morphology, phase identification, and functional groups of Ti-6Al-4V materials after anodization and silver plating in the field of bone implants. Through this study, the researcher hopes to provide deeper insights through the research question (RQ) of how variations in the anodization process and silverplating method can affect important characteristics of bone implants, such as morphological characteristics, phase groups, and phase identification. Thus, the results of this literature review will provide a solid foundation for the development of more effective and reliable bone implants in biomedical applications.

## 2. Methods

### 2.1. Overview

This study adhered to PRISMA (Pr andeferred Reporting Items for Systematic Reviews and Meta-Analyses) [23] and the guidelines established by Aromataris and Pearson's Joanna Briggs Institute [24] as depicted by Figure 1. The research topic and inclusion criteria were essentially the first steps of this investigation. The researcher then searched for, screened, and examined studies that met the requirements for inclusion. To give a thorough illustration of the subject being studied, the results of the research that were included in the qualitative analysis were combined and presented.

### 2.2. Research Question (RQ)

The research question is a key element that determines the direction and purpose of a scientific study in identifying knowledge gaps, developing theoretical understanding, and providing accurate information based on the results of previous research. The research question (RQ) in this study is related to the effect of variations in the anodization process of silver-plated titanium on important characteristics of bone implants, such as morphological characteristics, phase groups, and phase identification of bone implants. This RQ can be developed or presented in Table 1 below.

### 2.3. Inclusion Criteria

We looked for every study that satisfied the three inclusion requirements in order to respond to the research question. The type of evidence was the first criterion (Figure 1). In this criterion, researchers conducted a review with restrictions only on English and Indonesian articles published on Scopus, Web of Science, or found on Google Scholar with a time span of 2000 - 2023. To specifically answer the research question (RQ), the researcher applied the inclusion criteria of relevance and subject-focus of the evidence, where the researcher included research findings that focused on discussing the anodization process on silver-coated titanium and identified directly or indirectly on the findings regarding its effect on the morphological characteristics, phase groups, and phase identification of the resulting bone implants. The third inclusion criterion was quality evidence. As this study synthesized data from both quantitative and qualitative studies, we applied a generic quality criterion by including any article that had all the essential parts such as literature review, methodology, findings, references (thoroughness), explicit methods (explicitness), and clearly interpreted findings (clarity).



## 2.4. Search Strategy

For articles written in English, researchers first searched Scopus for all articles that included in their title, abstract, or keyword list the terms "anode", "anodizing", "titanium", and "silver plating". Then another search was conducted for articles that included synonyms in Indonesian on Google Scholar with the keyword "anodizing". After that, the researcher conducted a final search using the keywords "the effect of titanium anodizing parameters on material characterization". This search used Harzhing's Publish or Perish software and resulted in the identification of 1044 articles for eligibility testing.

### 2.5. Study Inclusion Characteristics (Screening)

In this stage, we identified articles that were suitable for qualitative analysis. Initially, we copied the titles of the 1044 articles to an Excel and Covidence (AI) sheet to compare and remove duplicates, reducing the number of articles to 958. Then, we excluded articles whose titles or abstracts indicated topics that were outside the scope of the study, reducing the number to 258 articles. After that, we read each abstract thoroughly and examined the 258 articles to exclude those that did not fit the inclusion criteria. In addition, we also excluded 232 article titles that did not contain words related to their effect on the predefined variables (e.g. physicochemical, biocompatibility, mechanical, corrosion, and antimicrobial). Furthermore, the researcher also did not take 8 article titles that were not openly accessible. As a result, 18 articles were identified that were eligible for analysis.

### 2.6. Analysis

Articles studying the effect of varying anodization parameters of silver-coated titanium on morphological characteristics, phase groups and phase identification are too heterogeneous in their reporting. Therefore, we used thematic analysis to synthesize, analyze, and report the findings of these articles. We followed the combined process suggested by Leavy [25] and Yin [26] which is commonly used. This process follows an inductive approach consisting of five steps: (1) data preparation and organization; (2) initial immersion; (3) coding; (4) categorization and theme formation; and (5) interpretation and conclusion.

After obtaining the final results of articles Figure 2. that were submitted for review after several selections, the researcher then used Microsoft Excel to conduct an analysis based on the coding done. A total of 18 articles were reviewed and analyzed to be able to classify the findings in synthesizing and answering the Research Question.



## 3. Results and Discussion

In this section, the researcher will present the results of analysis and synthesis to answer research questions related to how variations in the anodization process (electrolyte, voltage, and time) on silver-coated titanium can affect important characteristics of bone implants, such as morphological characteristics, phase groups, phase identification. The synthesis in the analyzed articles (can be seen in Table 2) is based on a predetermined year (can be seen in Figure 3).

Anodization	No.	Refs.	( In	Class Ifluei	ificat nce F	ion of indings	Description of Findings
Variations			P1	P2	P3	P4 P5	
Electrolytes in the Anodization Process	1.	[27]			~		The formation of nanoporous alumina layers (electrolytic) on the surface of aluminum can significantly affect its hardness. The anodization process enhances the surface hardness of aluminum by creating a dense oxide layer. In this case, the nanoporous nature of the alumina layer can further enhance hardness due to increased surface area and specific structure. The presence of regular pores can also contribute to surface mechanical properties, potentially increasing hardness compared to conventional anodization methods.
	2.	[28]	~				The main findings of this research include the influence of field strength on defect concentration in aluminum oxide films, the effectiveness of potentiostatic polarization in reducing defects, the negligible impact of polarization on film thickness, and the weak correlation between trap density and tunneling processes.
	3.	[29]		√			This study highlights the significance of oxide films on titanium for biocompatibility and corrosion resistance, as well as understanding the repassivation kinetics crucial for corrosion protection and preserving metals in various applications.
	4	[30]	~		1		The highest efficiency of the anodization process occurs in sulfuric acid solutions, resulting in the thickest oxide layer and highest surface hardness. In contrast, the process in phosphoric acid solutions exhibits the lowest efficiency, yielding the thinnest oxide layer with lower surface hardness compared to the base metal.
	5.	[31]	~				The main findings of the research indicate that the brightness of anodized aluminum layers is influenced by electrical current and processing time. The optimal brightness is achieved at 1 ampere and 10 minutes of anodization, whereas layer thickness is affected by 3 amperes and 30 minutes of anodization. This study demonstrates a clear relationship between anodization time on aluminum and both brightness level and layer thickness.
Voltage in the Anodizing Process	6.	[32]	~			V	<ul> <li>a. Anodized films on Titanium primarily consist of TiO<sub>2</sub> and Ti2O<sub>3</sub>, exhibiting a color spectrum from Blue-Yellow-Purple-Green.</li> <li>b. Films developed within the voltage range of 50-52V show the best corrosion resistance, being more compact, uniform, and having higher capacitance values compared to films developed at other voltage ranges.</li> <li>c. Anodized Titanium is commonly used in medical devices, orthopedic implants, dental implants, and aerospace industries due to its corrosion resistance and coloration effects.</li> </ul>
	7.	[33]	√			$\checkmark$	a. PEO (Plasma Electrolytic Oxidation) coatings significantly enhance the corrosion resistance of AZ61 alloy

Table 2. Summary of classification of research findings (P1: Physicochemical properties; P2: Biocompatibility; P3: Mechanical strength;

P4: Corrosion resistance; P5: Antimicrobial ability)

Anodization	No	Refs	C Int	Jassi fluer	ficati	ion o indin	ot σs	Description of Findings
Variations	140.	Ners.	P1	P2	P3	P4	<sup>53</sup> Р5	Description of Findings
								b. Thicker coatings formed on Mg-6Al-1Zn-1Ca and Mg-6Al-1Zn-2Ca alloys increase polarization
	_							<ul> <li>c. Excess calcium above 1% by weight in the alloy reduces both corrosion resistance and the ability to form apatite within PEO coatings.</li> </ul>
	8.	[34]	~			√	~	Sealing with beeswax and rosin significantly enhances the corrosion resistance of the anodic oxide layer formed on magnesium alloy AZ31. Sealed specimens demonstrate a substantial decrease in material loss and a corrosion rate five times lower compared to unsealed specimens. Beeswax and rosin effectively seal pores and cracks within the anodic layer, preventing penetration by
	0	[25]						corrosive solutions.
	9.	[22]	v				v	Ti6Al4V orthopedic implants using Electrochemical Impedance Spectroscopy (EIS). It was found that the thin, stable, and compact oxide layer obtained through potentiodynamic methods effectively protects the Ti6Al4V alloy. Additionally, after just 10 days of immersion in Simulated Body Fluid (SBF), uniform deposition of Hydroxyapatite (HAP) was observed, indicating potential to minimize dissolution processes and enhance interaction between bone and the implant.
	10.	[36]						Observations revealed significant negative effects of bending in the region with the highest tensile stress, as well as the regeneration of the passive layer in oxygen-containing media, enhancing the ability of the deformed surface layer to attract components forming bydroxyanatite.
	11.	[37]				✓		FE-SEM images reveal that the films become progressively thicker as the applied voltage increases. The thickness grows from 198.6 nm at 10 V to 435.5 nm at 20 V, reaching 1199 nm at 30 V. The observed color changes in the films are linked to variations in their thickness and pore density. The anodic film substantially boosts the alloy's corrosion resistance, improving it by nearly an order of magnitude. After anodization at 10, 20, and 30 V, the corrosion current density decreases in succession. The film serves as a protective barrier between the metal and the solution, with corrosion resistance improving as the film thickness increases. Anodizing Ti-6Al-4V combines the advantages of surface coloration and enhanced corrosion protection.
	12.	[38]	√					The main findings highlight the investigation into the phenomenon of breakdown in aluminum oxide dielectrics, with emphasis on the impact of polarity on breakdown voltage and the role of asymptetic charge distribution within the oxide
	13.	[39]						Anodizing voltage affects the color and thickness, with 50 V showing the lowest corrosion rate and a wider passive region in acid rain solution.
	14.	[40]	√			~		<ul><li>a. Chromaticity values vary with anodizing voltage, reaching the highest at 10 V and the lowest at 15 V.</li><li>b. Increasing anodizing voltage results in a decrease in the refractive index of the anodic film,</li></ul>
	_							<ul><li>indicating a decrease in film density, particularly noticeable above 55 V.</li><li>c. Anodizing in sulfuric acid solution significantly reduces the corrosion rate compared to non- anodized samples, with the lowest corrosion rate observed for samples anodized at 10 V.</li></ul>
	15.	[41]	~			√		The main findings include comparisons of surface morphology between titanium alloys anodized in different solutions, the corrosion resistance of Ti Beta-C alloy compared to other anodized alloys, and the effectiveness of electrochemical noise in describing corrosion behavior and surface integrity.
Time in the Anodizing Process	16.	[42]	~				√	This study demonstrates a proportional relationship between applied voltage and pore size in anodized aluminum oxide, highlighting the flexibility of anodized aluminum oxide for various applications.
	17.	[43]			√			The main findings relate to the comparison of anodic layers formed in various solutions, pore structure, surface roughness, and hydrophobic properties. The morphology of pore structure is highly dependent on the anodization time.
	18.	[43]			√			Surface modification of titanium, addition of nanosilicates to epoxy adhesives, exposure to high- energy radiation, and thermal durability significantly affect the adhesive bond strength of titanium.
Silver Coating	19.	[44]	√	√	√		1	This article discusses the historical use of silver for its antibacterial properties, various forms of silver utilized for antibacterial purposes, and its significance in consumer products and medical devices
	20.	[45]	√		√			The concentration of H2SO4 electrolyte solution influences the voltage and current output of used dry cell batteries containing H2SO4 solution and a 3 mM silver nanoparticle solution. The electrolyte solution concentration that maximally affects the output voltage of the used dry cell
	_							battery is 50%. Similarly, the electrolyte solution concentration that maximally affects the current output of the used dry cell battery is also 50%.
	21.	[46]	V					<ul> <li>This study demonstrates that:</li> <li>a. The optimal condition for the formation of single-phase nickel-cobalt oxide (NiCo2O4) is a Ni ratio of 33.3 mol%: 66.7 mol% with heat treatment at 400 °C.</li> <li>b. Pure spinel structure of NiCo2O4 was successfully synthesized with a cation ratio of 33.3 mol% Ni: 66.7 mol% Co at a calcination temperature of 400 °C.</li> </ul>
								c. The crystallinity of the prepared oxide increases with an increase in Ni content, while the crystal size increases with temperature.
	22.	[21]		~			1	The results of this study indicate that both silver-coated and zinc-coated nanotubes exhibit significant antibacterial properties, with the silver coating being more bactericidal compared to the zinc-containing coating. However, zinc oxide layers are more biocompatible than silver layers. Composite layers containing nano-silver and nano-zinc were successfully synthesized and tested for their antibacterial effects and compatibility with human cells. The final conclusion is that nano-
								silver remains toxic, whereas nano-ZnO layers are more biocompatible.

Anodization Variations	No.	Refs.	C	lassi	ficat	ion of		Description of Findings
			P1	P2	P3	P4	s P5	
	23.	[47]					√	This review critically summarizes the current understanding of the interactions between oil, oil dispersants, and sediments, their roles in developing oil spill response actions, and how these interactions may change in deep-water aquatic environments.
	24.	[48]						The findings of this study are: Engineered nanoparticles can induce oxidative stress through factors such as particle surface, size, composition, and the presence of metals, leading to reactive oxygen species (ROS) formation and subsequent toxicity. Understanding nanoparticle-induced oxidative stress is crucial as it can lead to genotoxicity, inflammation, and fibrosis. Comprehensive characterization of the physicochemical properties of nanoparticles is important for predicting and mitigating nanoparticle-induced toxicity.
-	25.	[49]	✓				✓	This study investigates the antibacterial properties of metal-containing nanoparticles, with silver nanoparticles (Ag NPs) demonstrating the strongest antibacterial activity compared to titanium dioxide and silica nanoparticles, as well as chlorhexidine. Ag NPs exhibit bacterial growth 25 times lower than chlorhexidine, making it the most effective disinfectant tested in this research. The findings suggest that Ag NPs could be a more effective alternative to chlorhexidine for infection control in dontistry.
-	26.	[50]						The main findings of the study include comparing the bactericidal activity of silver zeolite with silver nitrate against Escherichia coli cells, highlighting the involvement of silver ion transfer and the formation of reactive oxygen species in the bactericidal action of silver zeolite.
-	27.	[51]		√			√	Silver nanoparticles exhibit antimicrobial effects against bacteria and fungi, albeit with lower efficiency compared to silver ions, even at low concentrations in fabrics and coatings. Additionally, fiber absorbents coated with silver nanoparticles prevent biofilm formation during water treatment processes.
-	28.	[52]						A comprehensive overview of the history of silver in medicine, its clinical benefits, and its superiority as a broad-spectrum antimicrobial agent.
-	29.	[53]	√	√			√	Silver ion solution effectively reduces the number of bacteria, including S. aureus and E. coli, by more than 5 log 10 CFU/ml after 90 minutes of treatment. This study suggests that silver ions induce bacteria into a state called Active but Non-Culturable (ABNC), ultimately leading to their death. Electrically generated silver ion solutions induce bacteria into ABNC state, disrupting their ability to uptake and utilize substrates, causing morphological changes, and cell death.
-	30.	[54]	√	√			√	Silver nanoparticles exhibit effective bactericidal properties against E. coli, causing damage to the cell wall and accumulating on the bacterial membrane. This study concludes that silver nanoparticles have excellent antibacterial activity against E. coli, demonstrating potential as a new bactericidal material.
-	31.	[55]	1				√	Partially oxidized silver nanoparticles exhibit antibacterial activity, correlating with the level of chemically adsorbed Ag+ ions. Smaller particles show higher antibacterial activity based on equivalent silver mass content. Aggregation in high electrolyte media leads to loss of antibacterial activity, which can be prevented by complexation with albumin.
-	32.	[56]	V	✓			✓	The findings of this study indicate: The definitive molecular toxicity in silver nanoparticles (AgNPs) is from Ag+ ions, and the antimicrobial activity of AgNPs is solely due to the release of Ag+ ions. Morphological properties of AgNPs that influence antimicrobial activity primarily affect the release of Ag+ ions rather than providing direct particle-specific effects. The dose-response pattern of various AgNPs can be explained by the concentration of Ag+ ions released suggesting that there is no direct contribution of particle-specific effects to toxicity.
-	33.	[57]	V				1	The main findings of this study encompass the potential of silver nanoparticles as antimicrobial systems, the significant impact of surface properties on their efficacy, the role of Ag+ release in their mechanism of action, considerations of reactive oxygen species (ROS) formation as a mode of action, and the importance of understanding these modes of action to design effective antibacterial systems. Additionally, the paper highlights Ag+ as an antibacterial agent, the significance of oxidative dissolution in the antibacterial effects of nanoparticles, and the influence of chemical species on the actions of Ag NPs. The paper also emphasizes the high affinity of silver for various compounds found in natural media and the importance of controlling solution conditions during experiments.
	34.	[58]	√				√	This study explores the encapsulation of silver nanoparticles coated with PGA and ascorbic acid in PLGA microspheres to achieve a system with sustained antioxidant and antimicrobial activities. The synthesized particles are spherical with narrow size distribution, indicating successful encapsulation. This approach offers a promising pharmaceutical material with potential applications in infection treatment, particularly in orthopedic surgery and ocular drug therapy
-	35.	[59]	1				√	The main findings include the advantages of Electrophoretic Deposition (EPD) in ceramic production, its successful applications across various fields, as well as its simplicity, ease of use, and cost-effectiveness.



## **3.1. Titanium Anodization Process**

Anodizing is a method of surface modification of metals and their alloys that allows obtaining the desired properties of the oxide layer by electrode reaction in the metal combination driven by an electric field, and diffusion of oxygen ions on the oxide film formation on the anode surface. Anodization can be performed by controlling electrochemical parameters, such as electrolyte, voltage, current, time, and temperature [60]. Anodic oxidation can be performed potentiostatically or galvanostatically in different electrolytes [61]. The most used electrolyte solutions for anodization are H<sub>2</sub>SO<sub>4</sub>, Na<sub>2</sub>SO<sub>4</sub>, CH<sub>3</sub>COOH, H<sub>3</sub>PO<sub>4</sub>, and HF. The oxide layer formed by anodization differs according to the substrate, e.g. oxidation in chromic acid produces a TiO<sub>2</sub> layer on titanium substrates, but a layer consisting of TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>oxides on Ti-6Al-4V substrates [62].

The formation of the oxide layer during anodization involves a complex chemical mechanism, incorporating various fundamental chemical principles. The anodic layer is typically described as the outermost layer in contact with the electrolyte ( $H_2SO_4$ ), which contains  $OH^-$  ions, while the inner layer is identified as  $Ti(OH)_4$ . In this inner layer, the removal of hydrogen occurs, resulting in the formation of titanium oxide. The electrolyte concentration varies throughout the film, often expressed as  $TiO_2 \cdot xH_2O$ , comprising both the inner anodic oxide and the outer layer. At the positive terminal, the chemical transformations include:

The removal of oxygen and electrons from the titanium surface during the oxidation process.,

$$2\mathrm{Ti} \rightarrow 2\mathrm{Ti}^{4+} + 8\mathrm{e}^{-} \tag{1}$$

$$Ti (OH)_4 \rightarrow TiO_2 + 2H_2O \tag{2}$$

Terminals at negative polarity will develop hydrogen,

$$8H^+ + 8e^- \rightarrow 4H_2 \tag{3}$$

Combining the equations from (1) to (3), the overall process of oxide formation is as follows oxide is as follows:

$$8H^+ + 8e^- \rightarrow 4H_2 \tag{4}$$

By employing the anodization technique, titanium dioxide deposited on a titanium substrate predominantly exhibits the rutile phase of  $TiO_2$ . The resulting oxide layer offers enhanced corrosion resistance, with its thickness progressively increasing as the anodic voltage rises. However, the growth rate slows due to the formation of an oxide barrier that restricts current flow. During anodizing, hydrogen gas is released, and the titanium substrate heats up because of the chemical reactions and heat generated at the anode, as described by Eq. (4) [63].

Titanium anodizing provides several key benefits, including improved adhesion and bonding. Additionally, it enhances oxide thickness, which improves corrosion resistance, minimizes ion release, allows for surface coloring, and enables the formation of porous coatings. The structural and chemical characteristics of the anodic oxide layer can be significantly influenced by adjusting process parameters such as anode potential, electrolyte composition, temperature, current, voltage, and duration. Figure 4 illustrates a schematic representation of the anodizing process.

## 3.2. Effect of Electrolyte in The Anodizing Process (RQ1)

Electrolytes are materials in solution that carry an electric current with positive (cathode) and negative (anode) charges [64]. They are basic ingredients in the anodizing process to form a thick and stable oxide layer on metals and alloys to reduce corrosion. The electrolyte acts as an electronic barrier that separates the positive and negative electrodes [65]. In the anodizing process, the electrolyte is generally acidic, with the most used being sulfuric acid, chromic acid, phosphoric acid, and oxalic acid. The type of electrolyte can affect the oxide layer produced on the metal surface, including the hardness and thickness of the oxide layer. Furthermore, Hassel and Diesing [28] concluded that the thin oxide layer formed on titanium or aluminium metal in the anodizing process depends on the electrolyte and the increase in electrode potential change. Consistent with Sakairi et al. [29] showed that differences in electrolytes will cause differences in current density that occurs and electrode potential that occurs, so that it gives rise to different current efficiency numbers in each electrolyte.





In addition, Sihombing et al. [30] on the effect of electrolyte type in the anodization process on surface roughness and oxide layer thickness illustrates the influence of the electrolyte itself on the anodization process. Figure 5 shows the relationship between electrolyte type and oxide layer thickness in the anodizing process.

Figure 5 shows the relationship between the type of electrolyte and the thickness of the oxide layer in the anodizing process, showing that the highest oxide layer thickness in sulfuric acid solution reached a thickness of 13.88  $\mu$ m, followed by 1.43  $\mu$ m in oxalic acid solution, 0.41  $\mu$ m in chromic acid solution

and 0.28  $\mu$ m in phosphoric acid. It is also suggested that the oxide layer using chromic acid electrolyte, produces a thin oxide layer with a thickness of 0.02 - 0.1 mil (0.5 - 2.5 microns) and has the least effect on fatigue strength and is non-corrosive [31].

The type of electrolyte solution in the anodizing process also affects the surface hardness. Figure 6 shows the relationship between the type of electrolyte and the surface hardness in the

Figure 4. Schematic diagram of anodizing process for bone implants



Figure 6. Effect of electrolyte solution type on surface roughness anodizing process, indicating that the surface hardness of metals that have an oxide layer thickness above 1.0  $\mu$ m has a greater surface hardness than the hardness of the base metal, namely the product surface hardness of the metal in sulfuric acid is 287.9 HV, and in oxalic acid solution is 75.66 HV, while the surface hardness of the base metal is 66.75 HV. This proves that the oxide layer formed is both barrier and porous, that is, the barrier oxide layer has the characteristic of protecting the metal from corrosion attack, and the porous oxide layer and will

provide strong adhesive characteristics if the next layer is applied, or the metal surface is colored. Metals with an oxide layer thickness of <1.0  $\mu$ m have a surface hardness lower than the surface hardness of the base metal, namely the surface of the anodized product metal in chromic acid solution 64.7 HV and in phosphoric acid solution 41.3 HV. This occurs due to the process of dissolving Al into Al<sup>3+</sup> ions has a faster reaction rate than the rate of oxide formation that coats the metal surface and causes the metal to become thinner, so that the metal surface hardness becomes lower [30].

### 3.3. Effect of Voltage in Anodizing Process (RQ2)

Voltage is the potential difference between two points, which can be explained as the total work required to pass current from one point to another, measured in volt (V). The amount of voltage can affect the results of the anodizing process. Voltage plays a role in forming the resulting oxide layer, where the higher the voltage in the anodization process, the higher the potential difference that exists, so that the ionization energy increases. The increase in ionization energy results in an increase in the energy required to break the bonds of titanium ions. As a result, the number of ions from the electrolyte solution attached to the titanium surface also increases [32].

Studies by Anawati et al. [33] and Fitriana and Anawati [34] report a significant improvement in corrosion resistance due to the anodizing process. Corrosion, which leads to the release of metal ions, can impact both biocompatibility and mechanical integrity. When metal ions are released above tolerance levels, the osteointegration process can be inhibited [35]. Human body fluids contain aggressive chloride ions that can attack the natural oxide layer on metal implants. The anodizing process creates a similar passive layer on the metal surface, with the thickness of the oxide layer increasing as the applied voltage rises [36].

Figure 7 presents an optical microscope image showing the surface morphology of the anodic oxide film on Ti-6Al-4V. The anodic film exhibits a porous structure, as indicated by the presence of pores on the oxide layer. At 10V, the film displays a grainy structure with an average grain size of approximately 5  $\mu$ m (a). This grainy structure becomes less prominent, and the film densifies when anodized at 20V and 30V (b) and (c). The number of pores increases with higher anodizing voltage. The lowest pore density is observed in the coating anodized at 10V, while the highest pore density occurs at 30V. Films with fewer pores tend to absorb more light, resulting in a strong color appearance. Conversely, films with a high pore density appear lighter because the pores act as pathways for light waves. The high amount of reflective light from the anodic layer produces brighter colors. Additionally, the film thickness also influences the color appearance [37].







Figure 8. FE-SEM cross sectional of anodic film formed on Ti-6Al-4V alloy at [11]: (a) 10V; (b) 20V; (c) 30V Furthermore, Figure 8 shows the FE-SEM cross-sectional image of the anodic oxide film layer formed on Ti-6AI-4V. The film layer shows a uniform thickness over a customized observation area. The oxide layer formed at 10V voltage shows a thickness of about 198.6 nm. The films produced from 20V and 30V voltage were thicker by about 435.5 nm and 1199 nm, respectively. These results show that although the voltage affects the pore density, the applied voltage can also change the thickness of the film layer. In addition, the voltage will also affect the color. Based

on the findings of Saraswati et al. [37], the anodization process of the Ti-6Al-4V alloy generates different colors depending on the applied voltage. Similarly, it has been observed that increasing the voltage leads to a thicker anodic oxide layer [38]. This increase in the thickness of the film, in turn, causes a reduction in the refractive index [39]. The optimal thickness range of the oxide layer (5–20  $\mu$ m) ensures a balance between corrosion resistance and mechanical integrity, while excessive thickness can lead to internal stress or cracking. Furthermore, adhesion strength above 15 MPa is critical to withstand biomechanical stresses in implant applications, ensuring durability and functionality.

Table 3. Surface colors of Ti-6AI-4V alloy after anodization at various voltages



Furthermore, the voltage level influences the resulting color. As demonstrated by Saraswati et al. [37], the anodization of Ti-6Al-4V alloy at different voltages yields distinct colors, as summarized in Table 3 [41], At a low voltage of 10V, the anodic film appears gold. Increasing the voltage to 20V results in a dark blue surface, while anodization at 30V produces a light blue color. Transparent anodic films produce light interference that results in color variations depending on the reflective light wave [66]. Varying the anodization voltage changes the optical characteristics of the resulting anodic film. The resulting interference color depends on the number of pores and the thickness of the film [40].

## 3.4. The Influence of Time in the Anodization Process (RQ3)

The immersion time in the anodization process refers to the period from the beginning to the end of the anodization process. This immersion interval is a key factor that influences the outcome of the anodization process. A longer immersion duration will result in a thicker oxide layer, which enhances surface hardness. However, if the interval is too long, it can lead to excessive burning and erosion of the specimen.

The anodization time also plays a role in the formation of pores, where longer processes lead to larger pore diameters regardless of the applied voltage. This is caused by continuous current flow in specific areas, resulting in excessive heating in those areas [43]. Furthermore, Gómez-Méndez et al. [43] found variations in the surface of the anodic layer, with pores exhibiting different sizes and shapes. This can be illustrated through Figure 9.



#### Figure 9. Electron micrographs of anodic layers formed on Ti-6Al-4V 303K in different solutions [46]: (a) AHP 900 seconds; (b) AHP 1800 seconds; (c) CAA 900 seconds; (d) CAA 1800 seconds

Gómez-Méndez et al. [43] stated that the surface of the anodic lavers exhibits variations in pore size and shape. The morphology of the pore structure is highly dependent on the anodization time (Figure 9a and Figure 9b). A sponge-like porous structure was observed in the anodic layers formed at 900 and 1800 seconds. There was no significant difference in pore spacing, and the average pore diameter ranged between 200 and 300 nm. On the other hand, regularly spaced pores or nanotubular structures with a pore

diameter of approximately 43 nm were formed by the dissolution of the oxide on the  $\beta$ -phase grains of the Ti-6Al-4V alloy during 900 seconds (Figure 9c). However, when the anodizing time increased to 1800 seconds (Figure 9d), the pore or nanotubular structures were no longer clearly visible, possibly due to chemical dissolution.



Furthermore, the surface results of roughness the anodization varied with different time conditions as depicted in Figure 10. The average surface roughness of pure Ti-6Al-4V alloy is 2.75 µm. For samples anodized in AHP solution, the average roughness decreased with increasing anodization time from 3.62 to 2.73 µm. In contrast, for samples anodized in CAA solution, surface roughness increased with longer anodization times. Mechanical cross-linking is one of the key mechanisms for bonding

titanium alloys to other materials in aerospace applications [67]. Greater surface roughness provides a larger area for both mechanical and, occasionally, chemical bonding. The topographical features associated with increased roughness allow adhesives to penetrate more effectively, enhancing durability and bonding strength [68]. Thus, anodization conditions that produce rougher surfaces can improve adhesive bonding by creating a superior anchor profile. Researchers observed that the optimal roughness was achieved with anodic layers formed in CAA solution after 1800 seconds, yielding a roughness of  $3.92 \mu m$ , followed by layers formed in AHP solution after 900 seconds with a roughness of  $3.62 \mu m$  [43].

Prolonged anodization time can lead to potential trade-offs in material properties. According to research by Liang wu et al. [9], several potential trade-offs or compromises in the properties of Ti-6Al-4V alloy due to prolonged anodization duration exist. Firstly, excessive growth of the oxide layer: prolonged anodization tends to result in a thicker oxide layer, but excessive thickness can induce internal stress or even microcracks within the oxide layer. Secondly, excessive anodization duration can cause significant changes in surface structure, such as roughness, texture, or the ability to accept additional coatings. Thirdly, excessive anodization may lead to a decrease in the mechanical strength of the Ti-6Al-4V alloy due to negative effects on microstructure or defects in the oxide layer, such as residual stress formation or microcrack formation, which can reduce overall strength and toughness of the alloy. Therefore, it is crucial to carefully consider anodization parameters, including duration, to achieve an optimal balance between enhancing desired properties, such as corrosion resistance, and maintaining the necessary mechanical and morphological properties for specific Ti-6Al-4V alloy applications.

### Figure 10. Surface roughness

topography of anodic layers formed Ti-6Al-4V alloy at 303K in different solutions [46]: (a) AHP 900 second; (b) AHP 1800 second; (c) CAA 900 second; (d) CAA 1800 second

### 3.5. The Influence of Silver Coating After Anodization Process (RQ4)

Silver (Ag) is a metal commonly encountered in daily life. In ancient times, silver was used as a lining for barrels to transport wine on merchant ships. Silver possesses antibacterial properties. With the advent of antibiotics, the popularity of silver as an antibacterial agent gradually declined. However, in modern scientific advancements, silver has reaffirmed its crucial role in various fields. In today's era of modern medicine, silver nanoparticles (nanosilver) are utilized in treating various diseases caused by bacteria, fungi, yeast, and viruses, with concentrations in parts per million (ppm), which are considered safe for humans [45]. There are several methods for synthesizing silver nanoparticles, resulting in particles of various shapes such as spheres, disks, rods, cubes, prisms, rings, platelets, triangular prisms, and octahedrons, depending on the growth conditions [44]. Typically, spherical shapes are preferred for antibacterial nanosilver applications. One of the most common synthesis methods in solution is the chemical reduction method, involving silver precursors, reducing agents, and stabilizing agents [46]. Additionally, when growing silver nanoparticles, photoreduction using TiO<sub>2</sub> nanotubes is widely used along with several methods involving microorganisms [21].

A titanium oxide film layer is developed on a titanium substrate using the silver coating technique. This process is followed by a silver ion exchange reaction that embeds silver into the oxide surface. The primary goal is to enable the surface to release silver ions, which function as antimicrobial agents [69]. One of the techniques for silver coating is Electrophoretic Deposition (EPD), a colloidal process that utilizes the mechanism of electrophoresis where charged particles suspended in a liquid move under an electric field, causing them to deposit regularly onto the substrate surface, forming thin to thick layers [69]. Electrophoretic Deposition (EPD) can produce layers with precise composition, excellent adhesive strength, and thickness ranging from 1 to 500 micrometers. EPD consists of two stages: (i) charged colloid particles are driven towards an electrode by applying an electric field, and (ii) these particles deposit onto the working electrode to form a dense layer. This process is followed by drying and densification through sintering [59].

Electrophoretic deposition (EPD) stands out among other advanced forming methods due to its high adaptability, allowing it to be easily tailored for various applications [70]. For example, deposition can occur on flat, cylindrical, or differently shaped substrates with only minor adjustments to electrode design and positioning. While EPD is a wet process, it allows precise control over both the thickness and morphology of the deposited layers as a function of deposition time. As the deposition time lengthens, the deposition rate slows down because the electric field weakens during electrophoresis. Moreover, the layer thickness grows, and the concentration of suspended particles diminishes, further reducing the deposition rate over extended periods [59]. Figure 11 demonstrates the process of particle migration and deposition in suspension under the influence of an electric field [56].



Figure 11. Electrophoresis scheme and deposition of particles in suspension under the influence of an electric field Several studies have shown that the antibacterial effectiveness is influenced by factors such as nanoparticle size and distribution, the size and depth of nanotubes, and how particles are attached to the nanotubes [47]. The antibacterial properties are linked to the production of reactive oxygen species, the release of silver ions, and the internalization of silver nanoparticles [48]. An interesting feature of silver nanoparticles is their ability to prevent biofilm formation [49]. Numerous in-vitro studies have demonstrated the prolonged antibacterial effects of silver nanoparticles embedded in TiO<sub>2</sub> nanotubes [71]. However, the potential toxicity to human cells remains a concern, particularly due to the rapid initial release of silver from the coating [72].

In addition, silver has found a wide range of applications due to its antimicrobial properties [50], [51]. In the healthcare sector, silver is utilized in wound dressings, antimicrobial creams, and more recently, in biomedical implants. The antibacterial effect of silver is attributed to its ability to release Ag+ ions when it encounters water. These Ag+ ions are bioactive and can interact with proteins, amino acids, and receptors on bacterial cell walls. As a result, higher ionization capacity leads to a greater release of Ag+ ions, thereby amplifying the antimicrobial effects. Ag+ cations have a strong interaction with electron-donating groups containing sulfur, nitrogen, and oxygen [52].

The exact mechanism by which silver nanoparticles (AgNPs) exert antimicrobial effects is still not completely understood. Some studies suggest that the antibacterial properties of AgNPs are due to their similarity to ionic silver, accumulating on bacterial cell walls and causing damage that leads to cell death [53], [54]. Other research indicates that the antibacterial activity of AgNPs results from the release of Ag+ ions through oxidative dissolution upon contact with dissolved O<sub>2</sub> in water, which acts as an oxidizer [55]–[57]. The crucial role of O<sub>2</sub> in the oxidative dissolution of AgNPs has been confirmed in studies by Xiu et al. [56] and Lok et al. [55], comparing AgNP synthesis under anaerobic and aerobic conditions. Additionally, it has been found that Ag+ ions are closely associated with the surface of AgNPs [55], [73]. Further research also suggests that the size of AgNPs and the method of production, including the selection of reagents, impact their bioactivity



[55], [58]. However, there appears to be a general consensus that the release of Ag+ ions through oxidative dissolution is the primary mechanism of antibacterial action of AgNPs [53], [55]–[57], [74]. Figure 12 illustrates the discussed reaction pathways of AgNPs.

A study by Sondi & Salopek Sondi [54] investigated the inhibition of E. coli growth at various concentrations of AgNPs. Figure 13 shows the range of AgNP solutions tested, revealing that concentrations above 10 µg/ml (0.1 ppb) significantly inhibit bacterial growth. The research also inoculated E. coli with various concentrations of AgNP solutions found that higher and concentrations of AgNPs delayed bacterial growth for up to four hours under the tested conditions, after which bacterial growth resumed. The significance of these findings lies in their demonstration that AgNPs can effectively inhibit bacterial growth during the early stages of bacterial colonization.

## 4. Conclusion

Variations in the anodization process significantly influence the key characteristics of silvercoated titanium implants, shaping their physicochemical properties, biocompatibility, mechanical strength, and biological responses. This review highlights how electrolyte composition, voltage, and duration of anodization directly affect the micro- and nanostructures of oxide layers, which play a critical role in implant-tissue interactions and antimicrobial efficacy. These findings underscore the importance of optimizing anodization parameters to enhance implant longevity, stability, and integration with biological tissues, thereby addressing critical clinical challenges such as infection and corrosion resistance. In addition, the integration of silver coatings offers promising antimicrobial properties, further supporting the development of reliable and high-performance bone implants. Electrophoretic deposition (EPD) emerges as a versatile technique for achieving precise control over coating thickness and morphology, providing practical advantages over alternative surface modification methods. By emphasizing these advancements, this review offers actionable insights for researchers and practitioners aiming to improve implant functionality and patient outcomes. Beyond clinical implications, this study advocates for sustainable biomedical practices by highlighting how optimized anodization and coating techniques can minimize material waste and environmental impact. Such an approach aligns with the broader goals of advancing both innovation and sustainability in implant development, ultimately paving the way for safer, more efficient, and eco-friendly biomedical technologies.

## **Authors' Declaration**

**Authors' contributions and responsibilities** - The authors made substantial contributions to the conception and design of the study. The authors took responsibility for data analysis, interpretation, and discussion of results. The authors read and approved the final manuscript.

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