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تحضير وتوصيف طلاء متراكب نانوي من هيدروكسيباتايت- اوكسيد معدن على سبيكة Ti13Nb13Zr بتقنية الترسيب الكهربائي للتطبيقات الطبية الحيوية

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Preparation and Characterization of Hydroxyapatite-Metal Oxide Nanocomposite Coating on Ti13Nb13Zr Alloy by Electrophoretic Deposition Technique for Biomedical Applications

A thesis

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1.1. Introduction

Biomedical alloys have become indispensable components in the delivery of cutting-edge healthcare solutions. These specialized materials have revolutionized the field, serving as the foundation for a wide range of medical devices, implants, and therapeutic applications that have significantly improved the quality of life for countless individuals [1]. These significant materials, composed of carefully selected metallic elements, illustrate an individual grouping of, chemical, biological, and mechanical, characteristics that make them indispensable in various biomedical applications [2].

Biomedical alloys have unique properties that make them ideal for use in the medical industry. Their exceptional strength, and biocompatibility corrosion resistance enable to development durable & long-lasting medical products that can withstand the human body's demanding environments.[3][4]. However, Metal implants were rarely implanted before the introduction of the sterile surgical Lister method in the 1865s; for example, Steeles, gold, or silver spinal cables and bone pins were efficient [4].

Metals have now found widespread use in orthopedics, becoming an integral part of most orthopedic implants. Bone plates, pins, and screws are examples of temporary tools in this category, whereas total joint replacements are examples of permanent implants and engineered to exhibit excellent biocompatibility, ensuring compatibility with the human body [5]. All Biomedical materials especially biomedical alloys undergo rigorous testing to assess their interaction with biological systems and minimize adverse reactions [6].

Furthermore, these alloys possess remarkable corrosion resistance, safeguarding their structural integrity and preventing releasing potentially harmful ions of metallic implants in the human body. This corrosion resistance remains crucial for the long-term reliability and functionality of bioimplants [7] Investigation of biomaterials metallic for nontraditional application in the reconstruction of hard tissues and organs has gained momentum in recent years [8].

Although a wide range of metals and alloys are potentially producible in industrial settings, only a small percentage of these materials actually implant materials that are biocompatible and have the other features they need to serve their purpose for a long time [9]. Most orthopedic medical devices on the market now include the form components [10].

According to the principal alloying element, the materials may be divided into one of four separate types, Stainless steels, alloys based on cobalt, alloys based on titanium, as well as a variety of additional alloys (such as NiTi and alloys of magnesium and titanium)[8] [10]. Several medical implants have received approval from the United States Food and Drug Administrations (FDA) making of metallic components that fall into the first three classifications[11].

In addition to that, the discipline of orthopedics makes frequent use of these different gadgets. Figure1-1 depicts typical clinical application scenarios. Due to their unique material properties [12].

Biomedical alloys find extensive use in orthopedic applications, where their mechanical strength and compatibility with bone tissue are paramount. Alloys of Titanium and titanium element, such as TNZ, are generally working in loadbearing implantation, including joint replacements, bone plates, screws, and dental applications [13]. These alloys offer a favorable combination of strength, low density, and corrosion resistance, enabling implants to withstand mechanical stresses and promote osseointegration, the implant is integrated with the surrounding tissues of bone [14][15].

Nevertheless, elastic modulus reduction between human bone tissues and implantation materials results in a phenomenon known as the "stress shielding effect" during the implantation process [16]. The " effect of stress shielding " occurs when a biomedical implant, such as a titanium alloy, is placed inside the body. It refers to the reduction in stress and strain on surrounding tissue bone produced because the elastic modulus of the implant material and the bone do not match [17]. Consequently, this can result in implant loosening, possibly leading to implant failure [18].

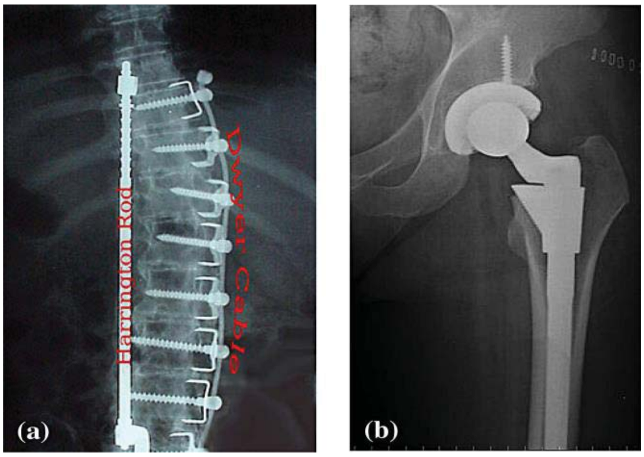

Figure 1-1: a. Harington rod is a stainless-steel surgical implant, b. the stem of total hip replacement is typically made of titanium-based alloys [19]**.**

Therefore, the investigation of novel implant materials is of the utmost importance if these issues are going to be resolved and the success rate of implantation surgery is going to be increased. Implantable materials have made significant contributions to contemporary medicine [20]. Because of the necessity for implants that are extremely resistant to corrosion, biocompatible, and able to bind to bone during Osseointegration, the human body offers materials scientists a particularly tough environment to conduct their research [21]. Implants covering all areas of usage in the human body, including as orthopedic, spinal, dental, cardiovascular, neurological, and urological applications, have made use of metallic biomaterials in the past, and they will continue to make use of these materials in the future [17], [22]. However, the most important aspect of the safe use of metallic implants is investigation problems of material, such as corrosion resistance and biocompatibility [23]. Orthopedics places a premium on mechanical performance in load-bearing locations, especially under biological working circumstances [24][25]. A titanium implant's roughened surface aids Osseointegration by promoting osteogenic activities such as cell adhesion, cell proliferation, protein adsorption, and calcium deposition [26].

Coatings made of ceramic metal oxides, which are biologically compatible with the human body, have been shown to lessen the negative effects of replacement alloys, which are distinguished by their high adaptability in the body of the organism [27]. Furthermore, these ceramic materials will prevent or, at the very least, greatly lessen the defects that may appear in the one or two decades of the existence of the replacement implant [28].

The introduction of titanium implants into bone structures results in the same kinds of healing responses as trauma [29]. The breakdown and rebuilding of bone tissue happens in tandem. Osseointegration's remodeling phase is ongoing [30] [31]. As a consequence, bone reconstruction is a field that is always evolving to meet the needs of its patients [32].

Histological examination has revealed that modified titanium surfaces can produce more bone than pure titanium surfaces during the early stages of osseointegration [33]. Studies investigating the effects of surface modifications on bone response to dental implants have demonstrated this [34].

1.2. Biocoatings

Biocoatings have emerged as a promising strategy for enhancing performing and biocompatibility implant surfaces on various applications related to biomedicine [35] [36]. These coatings, typically composed of biocompatible materials or bioactive compounds, are applied onto the surface of implants to modulate their interactions with the surrounding biological environment [37].

It explored the importance of bio-coatings, their benefits, and the different types of coatings used to improve the biocompatibility and functionality of implant surfaces [38] [39]. Deposition of nanoparticles such as calcium phosphate and nanometal oxides coatings is primarily responsible for an osteoblast's proliferation or the establishment of a robust implant bone connection capacity [40] [41]. In most cases, these coatings are either hydroxyapatite (HA)with ceramic materials such as (MgO, YSZ, and Al_2O_3) are substituted with additional elements [42] [43]. The scientific Problem this Research is that TNZ alloy is a material have been used in biomedical applications, such as implants. This alloy has many advantages, for example, unlike other titanium alloys, it does not cause cell death and has a lower modulus of elasticity[44]. There are also some problems associated with it. Here are some of the problems of TNZ alloy such as Surface deterioration: Changes in surface topography may result of the TNZ alloy, which may subsequently lead to a decline in the mechanical characteristics of the alloy [45], Cytotoxicity: One of the most challenging things about giving TNZ alloy antimicrobial properties is doing so without making it toxic. This can happen when one of the alloy elements reacts with various compounds in the body, such as the reaction of the element niobium in the body to form toxic compounds, which may lead to damage to bone cells [46]. In addition, the Homogeneity of coatings: The uniformity of nanocoating, when combined with certain Nano oxide coatings on the TNZ alloy, may be altered by the composition of the suspensions, voltage, and concentrations used during electrophoretic depositions [47]. The TNZ alloy exhibits several advantageous properties that render it suitable for various biomedical applications. However, to fully maximize its performance, some problems must be handled effectively.

Titanium alloys, namely the TNZ type, are widely regarded as the predominant material for implant production in orthopedic applications. Human body fluids may corrode metals and other materials, leading to the release of metal ions.

This has happened to TNZ alloys, which exposes them to the element zirconium; this might cause harm to the surrounding tissue and may have harmful effects if the passivation layer in the TNZ alloy fails. It's a significant worry that the tissue causes discoloration, cell poisoning, pain, and joint failure, all of which rule out its use in various biomedical contexts. Limited research has explored the electrophoretic deposition (EPD) technique for covering TNZ alloy implants with nanocomposite materials. Studies have explored parameters like applied voltage, deposition time, and concentration using biocomposite materials like hydroxyapatite, magnesium oxide, YSZ, and alumina. Most studies focused on physical properties, roughness, antibacterial properties, wettability, and corrosion resistance. However, few have explored multilayers of these materials or the EPD approach for composite coatings.

1.3. Literature review

M. Farrokhi et al (2018) [48]: The research shows that hydroxyapatite nanoparticles structured like fibers and titania suspensions in an ethanolic medium form nanocomposite coating on TNZ surface alloy. Coatings with varying titania/HA particle concentrations were produced by electrophoresing the suspensions. The finishes, applied with FHA at a concentration of 50 wt.% or higher, did not develop any cracks when drying. Coatings containing 50 and 75 weight percent FHA demonstrated superior corrosion resistance when tested in liquids simulated body fluid (SBF).

B. Mishal et al. (2018) [47] Coating thickness, adhesion, corrosion resistance, wettability, microhardness, and solution composition vs electrophoretic deposition voltage were investigated in this work. At 15, 30, or 50 V, hydroxyapatite at concentrations of 0.1, 0.2, or 0.5 mg nano-HA in a 100 mL solution was electrophoretically applied to the TNZ alloy. Using polarization curves, scanning electron microscopy, and atomic force microscopy to assess corrosion, nano-indentation, nano-scratch, and contact angle evaluations in synthetic body were performed. Both process factors have complicated and linked impacts about the make-up and features of HA coating, due to the shape, size, and contents of hydroxyapatite particles in suspensions. The deposition parameters of 0.1-0.5 mg/100 mL nanohydroxyapatite and 15-50 V EDP voltage result in homogenous, porous, hydrophilic, and less corrosion-resistant coatings (2-29 μm thickness) compared to titanium alloy.

L. Sorki et al. (2019) [49] : In this research, hydroxyapatite (HA)-chitosan-titania nanocomposite coatings were formed on 316 L stainless steel using electrophoretic deposition (EPD) from alcoholic (methanol and ethanol) suspensions containing 0.5 g/L chitosan and 2 and 5 g/L HA and 2 and 5 g/L Titania. The effect of different parameters on the deposition rate, morphology, and corrosion resistance of the coatings in simulated body fluid (SBF) at 37 °C has been studied. The coatings' properties were investigated by Fourier infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). Based on the results of this work, it was found that the deposition rate in ethanolic suspensions is lower than in methanolic ones. Moreover, the coating surface was smoother when the ethanol was used as a solvent in suspensions in comparison to the ones where methanol was the solvent. The coating deposited from a suspension containing 0.5 g/L chitosan, 2 g/L HA, and 5 g/L titania with ethanol as solvent had the highest corrosion resistance in SBF at 37 °C.

N. Nuswontoro et al. (2019) [50]: Electrophoretic Deposition (EPD) has been chosen as the coating method because of the simplicity of the making, the relatively low cost, and the ability to coat things with complicated designs. In this method, electrical current is used through the cathode and anode within the HA suspension. The electrical potential (voltage) will transport the small particle of HA to be deposited on the surface of TNZ until producing an even layer of HA coating. This coating process requires two major parameters, which are voltage and coating times. The desired quality of HA coating that would be produced can be achieved by adjusting these parameters. Voltages are in the range of 7, 10, and 13 volts, while coating times are in the range of 7, 10, and 13 minutes. Based on the result, it is known that the best HA coating that can be produced is at 10 minutes and 10 volts. This best result shows good surface morphology, optimum value of mass growth, coating thickness, and surface coverage. Based on this research, it can be concluded that increasing voltage and coating times will increase the coating thickness and surface coverage of HA coating. This result shows that the EPD can be used to produce a TNTZ titanium implant that is coated with HA for orthopedic applications.

M. Bartmański et al. (2019) [51]**:** In this research, hydroxyapatite coatings are used for surface modification of long-term implants. These coatings are characterized by high thickness and poor adhesion to the metallic substrate. The present research is aimed at characterizing the properties of (Nano HA) with the addition of copper nanoparticle (Nanocpper) coatings deposited on the Ti13Zr13Nb alloy by electrophoretic deposition. The deposition of coatings was carried out for various amounts of Nano Cu powder and various average particle sizes. Microstructure, topography, phase, and chemical composition were examined with scanning electron microscopy, atomic force microscopy, and X-ray diffraction. Corrosion properties were determined by the potentiodynamic polarization technique in simulated body fluid. Nanomechanical properties were determined based on nanoindentation and scratch tests. The wettability of coatings was defined by the contact angle. It was proven that HA coatings containing nano copper, compared to HA coatings without nanometals, demonstrated a smaller number of cracks, lower thickness, and higher nanomechanical properties. The influence of the content and the average size of nano cu on the quality of the coatings was observed. All coatings exhibited hydrophilic properties. The deposition of HA coatings doped with nano copper may be a promising way to improve the antibacterial properties and mechanical stability of coatings.

S. Sarabjeet et al. (2021) [52]**:** They discussed the research on β-Ti alloys in the medical field, focusing on design, biological responses, strengthening mechanisms, and low-cost implants with high biocompatibility. It highlights the importance of synchronizing β stabilizer content and using tailored thermo-mechanical techniques for low-modulus-high-strength implants. The paper also highlights the evolution of patents in this field from 2010 to 2020. It presented the design and development of β-Ti alloys and addressed their capabilities to obtain properties consistent with the target implant applications.

N. Ayad et al. (2021) [53] **:** The electrophoretic deposition (EPD) characteristics of hydroxyapatite powder on T4V6Al alloy bone implant materials were examined in this work. Various currents were tested. Coating layer characterization, surface roughness, and thickness are the primary areas of study. The results demonstrate that compared to uncoated Ti, coating with AC EPD increases grain density and hydrophilic contact angles. The research shows that materials for bone implants should be resistant to corrosion and biocompatibility.

R. Dorota et al. (2021) [54]**:** in this research Three coatings suitable for biomedical applications, including the dispersion coating composed of multi-wall carbon nanotubes (MWCNTs), MWCNTs/TiO2 bi-layer coating, and MWCNTs-Cu dispersion coating, were fabricated by electrophoretic deposition (EPD) on Ti Grade II substrate. Scanning electron microscopy, energy-dispersive X-ray spectroscopy, and nanoindentation were applied to study topography, chemical and phase composition, roughness, hardness, Young’s modulus, plastic, and elastic behavior. The results showed that the best mechanical properties in terms of biomedical application were achieved for the MWCNTs coating with a titania outer layer. Nevertheless, both the addition of nano copper and titania improved the mechanical resistance of the base MWCNTs coating. Compared to our previous experiments on Ti13Nb13Zr alloy, a general tendency is observed to form more homogenous coatings on pure metal than on the alloy, in which chemical and phase compositions are more complex.

P. Bansal et al (2021). [55]**:** This research looked at the possibility of covering bone implants with strontium-reinforced hydroxyapatite (HA-Sr) by plasma spraying in order to increase their corrosion resistance and mechanical strength. In comparison to titanium substrates coated with pure HA, the results demonstrate improved clinical performance, stronger corrosion resistance, and a harder surface as the Sr percentage increases. This might have positive implications for the use of bone implants in the future.

L. Mostafapour. et al. (2021) [56]: examined the kinetic model that characterized the time-dependent change in deposition yields for electrophoretically created nanocomposite coatings of yttria stabilized zirconia (YSZ) and aluminum oxide (Al_2O_3). Sintering was aided with aluminum powder, which also provided the raw material for the creation of aluminum oxide (Al_2O_3). The solvent used was a combination of ethanol and acetylacetone. Analysis of pH, zeta potential, and particle size allowed for the evaluation of interactions between electrostatic charges on surfaces and in suspensions, which were crucial in the preparation of a stable suspension. Using three evenly distributed solutions, comprising nano-structured Yttria in 50, 30, and 0 wt. percent of Al nanoparticles, Successful electrophoretic deposition (EPD) was achieved. The EPD kinetic at 0 to 300 second was evaluated and measured by applying the Baldisseri formula, taking into account three distinct voltages that are used (20, 40, and 60 V), depositions duration, coupled with the percentage by weight of Aluminum particles. It was demonstrated that the experimental data and the findings obtained from the Baldisseri model were almost identical. Deposition rate decreases as YSZ nanoparticle concentration and duration rise, according to the results. The last procedure was to dry and sinter the coated substrates at 1150 °C. Using scanning electron microscopy, surface morphology and coating quality were investigated throughout the (FE-SEM).

S. Rahmadani et al. (2022) [57] A bioactive hydroxyapatite (HA) coating was applied to a commercially pure Ti surface using electrophoretic deposition (EPD). The coating structure was optimized to achieve a stable layer and optimal corrosion protection. The coating was deposited at constant voltages of 20, 30, and 40 V for 30 minutes in a HA/DMF suspension. The layers, consisting of HA grains with a Ca/P ratio of 1.82, gradually increased in thickness with formation voltage. The suitable coating thickness for biomedical applications was at least 50 µm. The high compaction of HA grains at 30 V resulted in higher polarization resistance and lower corrosion current density.

L. Julia et al. (2022):[58] this studied aims to examine the impact of physicochemical properties of surface layers on implant processes made of titanium biomaterials in bone structures. The study also evaluates the

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تركزت هذه الدراسة على تطوير طلاء مركب للأجهزة الطبية باستخدام تقنية الترسيب الكهربائي، حيث يتكون الطلاء من مزيج من المواد النانوية غير العضوية وهي الهيدروكسي أباتيت HA، أوكسيد المغنيسيومMgO - ، الزركونيا المستقرة باليتريا YSZ ، وألومينا Al_2O_3 بالإضافة إلى الكيتوزان. تم اختيار سبيكة Ti13Nb13Zr ، وهي سبيكة طبية قائمة على التيتانيوم، لمعالجة سطحها لأنها تتمتع بخصائص ميكانيكية متوافقة ومع قابلية حيوية ممتازة. كان الهدف من هذه الدراسة هو تحسين خصائص سطح Ti13Nb13Zr من خلال مقاومته للتآكل، واستجابته البيولوجية، مما يجعلها أكثر ملاءمة لتطبيقات زراعة الأجهزة الطبية. تم اختيار هذه المواد بناءً على توافقها الممتاز مع البيئات البيولوجية وقدرتها على تحسين الخصائص الميكانيكية للطلاء، مما يضمن تكيفها ومرونتها عندما تستخدم كبدائل زراعية طبية.

تم تقسيم البحث إلى ثلاثة أقسام رئيسية. في القسم الأول، تم تصنيع طلاء نانوي يتكون من الهيدروكسي أباتيت (HA) وتم ترسيبه بتركيزات مختلفة (2، 4، 6 غرام/لتر) على سطح سبيكة Ti13Nb13Zr .تم تحديد أن التركيز الأمثل هو 4 جرام/لتر، حيث أعطى أفضل الخصائص من حيث أداء الطلاء. في القسم الثاني، تم إضافة أكسيد المغنيسيوم (MgO) إلى طلاء الهيدروكسي أباتيت بنسب وزن مختلفة من HA85MgO، HA10MgO، HA15MgO . تم اختيار العينة المطلية بـالطلاء المركب النانوي HA15MgO كأفضل عينة. اما في القسم الثالث، فقد تم استخدام احصاء تاجوتشي لاختيار الظروف المثلى لعملية الترسيب، مع مراعاة عوامل مثل الجهد والتركيز ووقت الترسيب. تم اختبار كل من هذه العوامل على ثلاثة مستويات مختلفة لتحديد أفضل ظروف تكوين الطلاء.

بعد هذه الخطوات لتحسين الطلاء، تم اختيار أفضل طلاء مركب يتكون من HA، Al_2O_3، MgO و YSZ بناءً على معايير الأداء مثل الخشونة، السماكة، قوة الالتصاق، وزاوية التلامس. أظهرت نتائج تحليل حيود الأشعة السينية (XRD) للعينات غير المطلية والمطلية لسبيكة Ti13Nb13Zr ان الطلاءات أظهرت هيكلًا بلوريًا متعدد الأوجه. تم تقدير حجم البلورات باستخدام معادلة شيرر، وكان الحجم المتوسط للبلورات يتراوح بين 6.35 نانومتر و 44.10 نانومتر لجميع الطلاءات، مما يدل على تكوين الجسيمات النانوية بنجاح. أكدت نتائج التحليل الطيفي للأشعة السينية (EDX)التركيبة الكيميائية للطلاء، بينما أظهرت صور المجهر الإلكتروني الماسح (SEM) أن الجسيمات النانوية تحتوي على أحجام حبوب غير متجانسة وأشكال غير منتظمة، وكانت أحجام الحبوب للطلاءات المركبة النانوية تتراوح بين 31.86 نانومتر و 107.68 نانومتر. تم استخدام الميكروسكوب الذري (AFM) لقياس خشونة سطح سبيكة Ti13Nb13Zr المطلية، حيث كانت قيم الخشونة Ra تتراوح بين 23.99 نانومتر و 105.30 نانومتر، وكانت قيم الخشونة الجذرية

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المتوسطة (RMS) تتراوح بين 0.32 نانومتر و 0.66 نانومتر، وكانت أحجام الجسيمات تتراوح بين 42.21 نانومتر و 69.42 نانومتر.

لتقييم الخصائص الميكانيكية والبيولوجية للطلاءات، تم إجراء اختبارات حيوية باستخدام الأرانب النيوزيلندية بالإضافة إلى الفحوصات المختبرية. أظهرت النتائج أن الطلاء المركب ($HA15MgO15YSZ15Al_2O_3$) كان خاليًا من الشقوق، ومناسبًا حيويًا، ومتماسكًا تمامًا. أظهرت نتائج تحليل XRD و SEM تكوين طبقة جديدة من الهيدروكسي أباتيت، مع ملاحظة تغييرات في قمم أطوار الهيدروكسي أباتيت بعد اختبار النشاط البيولوجي في سائل الجسم المحاكي (SBF) لمدة أسبوعين، مما يدل على النشاط البيولوجي للطلاء في البيئة البيولوجية.

تم إجراء اختبار تآكل كهروكيميائي لقياس الجهد المفتوح للعينات غير المطلية والمطلية في سائل (SBF) عند درجة حرارة 37° .أظهرت العينة غير المطلية جهدًا مفتوحًا قدره (-0.50 فولت)، والذي تحول إلى جهد أكثر نبلاً في العينات المطلية، مما يشير إلى استقرار أفضل ومعدلات تآكل أقل. أكدت هذه النتائج أن الطلاء المركب حسّن بشكل كبير مقاومة التآكل، كما تم التأكد من انخفاض معدل التآكل للعينات المطلية مقارنة بالعينات غير المطلية.

تم أيضًا دراسة النشاط المضاد للبكتيريا ضد بكتيريا E. coli و Staphylococcus.أظهرت نتائج الفحص أن الطلاء المركب لديه مقاومة جيدة، مع زيادة معدل التثبيط للبكتيريا، مما يدل على فعالية الطلاء في مقاومة نمو البكتيريا. في الاختبار الحي، تم زرع عشرة أرانب نيوزيلندية مع زرع براغي غير مطلية ومطلية في عظام الساق، مع براغي مطلية وغير مطلية في كل ساق. تم قياس عزم إزالة الغرسات بعد شهرين، مما أظهر تكاملًا أفضل بين العظم والغرسة مع الغرسات المطلية.

في الختام، أظهر الطلاء المركب النانوي ($HA15MgO15YSZ15Al_2O_3$) على سبيكة Ti13Nb13Zrمقاومة فائقة للتآكل، خصائص مضادة للبكتيريا، وقوة ارتباط عالية بالعظام مقارنةً بالطلاءات الأخرى. أظهر الطلاء أداءً ممتازًا من حيث الخصائص الميكانيكية والبيولوجية، كما هو موضح من خلال الاختبارات في المختبر والاختبارات الحية، مما يجعله مرشحًا واعدًا لاستخدامه في الأجهزة الطبية، مما يحسن من متانتها وكفاءتها في الجسم البشري.