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Effect of Coating Time of Electrophoretic Deposition of Hydroxyapatite-Chitosan-Magnesium Oxide Coatings on Ti-6Al-4V Alloys

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ABSTRACT

This research aims to evaluate various coatings on titanium alloy substrates to increase corrosion resistance and surface quality. This involves coating the substrates with layers of Hydroxyapatite (HAP), Chitosan (CS), and Magnesium oxide (MgO) using the EPD technique. The cell voltage value was set to 5 V, while the coating time was set to 5, 10, and 15 min. SEM was used to measure the thickness of the cross-sectional coating layers, and it was also used to show the morphology of the composite coating. From the results 10-minute deposition time has been established as the most optimal to produce an even, thick, uniform, and dense coating with good mechanical properties. The thickness more than doubles with increases in time with the coat almost tripling at 15 minutes and 5 minutes respectively. EDX analysis showed that the elemental compositions of calcium, oxygen, and phosphorus increased after 15 minutes, indicating a dense coating. The research findings indicate that the adhesion force transitions to a marginally positive value of 0.05 nN, suggesting a weak attachment rather than a robust bond. Additionally, Young's modulus shows a significant decline to 3.93 MPa, which signifies a reduction in the stiffness of the material. The surface stiffness decreases to 0.19 nN/nm between 10 and 15 minutes, indicating a loss of rigidity. Although longer coating times may increase thickness, they can also reduce functionality and raise defect risks. This reinforces that electrophoretic deposition (EPD) effectively improves the alloy's surface quality for biomedical applications.

1. Introduction

Two primary parameters control the EPD process: Charged particle mobility is influenced by suspension parameters, including Zeta potential, pH, solvent viscosity, liquid dielectric constant, and conductivity. Processing factors include deposition time, voltage, substrate conductivity, and material concentration [1, 2].

Titanium alloys, especially Ti-6Al-4V, are well known for their superior mechanical properties, biocompatibility and corrosion resistance all of which lead to their use in the construction of biomedical implants [3-7]. Nonetheless, despite these advantages, titanium alloys have limitations like low bioactivity, low Osseointegration and poor wear and corrosion resistance over time. These limitations can lead to implant failure, highlighting the importance of improving the surface properties even more[8, 9]. The bioactivity and integration with surrounding bone tissue can often be improved by performing a surface modification of the Ti-based alloys. One of the most effective methods relied on the use of different bioactive coatings such as hydroxyapatite. Chitosan is a natural

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polysaccharide from chitin and is biodegradable, biocompatible, and exhibits antibacterial characteristics. It is possible to develop chitin-based modifications on the surfaces of titanium that promote cell adhesion as well as cell proliferation [10, 11].

Powder hydroxyapatite (HA) can be considered a kind of calcium phosphate which resembles more closely to the mineral content of the bone. However, when applied as a coating hydroxyapatite increases the osseointegration of the implants as it provides a good substrate for bone cell propagation[12, 13]. Moreover, HA coatings can increase Ti-based alloy corrosion resistance, which also prolongs the implant service life. Ilhamdi et al[14], improved the bioactivity and performance of the Ti-6Al-4V ELI alloy by applying a bilayer hydroxyapatite (HA) coating. The hydroxyapatite coating provided the additional advantage of improving tissue fixation, preventing the implant from corrosion, as well as allowing for good adhesion that overcame in part low bioactivity of the alloy and improved its long-term applicability in biomedical implants.

The benefits of these two materials are maximized by applying chitosan and hydroxyapatite composites on Ti-based alloys. While chitosan is effective as a supportive polymer, hydroxyapatite is responsible for providing the bioactivity required for osseointegration. Such a composite coating enhances the biocompatibility and bioactivity of the titanium alloy while enabling controlled drug release, increasing healing chances and lowering post-surgery infection risk [15]. Elham et al[16], synthesized Chitosan-Bioactive Glass (CS-BG) Nano composite coatings on Ti-6Al-4V alloy components for improved properties. By varying the BG content (0.5, 1, and 1.5 g/L), they observed that the more the BG was increased the better the adhesion strength and the bioactivity, corrosion resistance, surface roughness, and wettability. Out of all the BG content used, the 1.5 g/L BG coating was the most effective due to the appearance of constant BG distribution and increased coating thickness. In vitro studies further proved the overlay to be effective when more bone apatite layers built up and good cell adhesion occurred. This showed that the CS-1.5 g/L BG significantly enhances the performance of the alloy.

In the last few years, considerable interest has emerged, regarding the use of electrophoretic deposition (EPD) as a surface treatment for titanium alloys in particular Ti-6Al-4V, to improve their biological and mechanical properties for use in biomedical implants. Specifically, Xi Chen et al [17], have shown that there is a remarkable improvement in the corrosion resistance of titanium alloys when a continuous graphene film is coated via EPD where the open circuit potential was measured at -0.01 V and the corrosion current density was reduced to 40% of that of the uncoated alloy. Likewise, Hamil et al [18], EPD to apply titanium dioxide (TiO_2) films onto titanium alloy Ti-6Al-4V against a corrosion rate of 2.970 × 10−4 mm/y, In their study, Juli admi et al [19], applied hydroxyapatite coatings derived from bovine bone onto a titanium alloy surface. This process resulted in 95.89% surface coverage, with a particle mass of 6.97x10^3 µg using 25 µm particles. Eshghinejad et al [20], also illustrated the efficiency of bio composite coatings with BG-GO including the 50 to 55-micron core bio composite mosaic coatings showing the greatest corrosion resistance as well as antibacterial activity. EPD was further demonstrated by Affi et al [21], to be viable for the fabrication of hydroxyapatite and TiO_2/HAP coatings with better surface properties of 0.88 Ra and micro-hardness of 1024 HV respectively.

Li-Chong Xu et al, [22] discuss the Adhesion forces between latex microspheres and glass beads on a surface under different ionic strengths . The study found that the negative values for adhesion force (e.g., -0.49 nN for latex microspheres and -0.67 nN, mean the force needed to detach the microsphere from the surface. A more negative value means stronger adhesion. For the glass beads, the adhesion forces are less negative (e.g., -0.25 nN and -0.42 nN) which means weaker adhesion than latex microspheres. Latex microspheres have higher adhesion forces at all ionic strengths than glass beads. So there are stronger interactions between latex microspheres and the surface.

Adhesive Forces The interplay between mechanical interlocking and chemical bonding at the interface affects adhesive strength, Understanding the adhesive forces can help in optimising the composition and deposition parameters for improved performance. These composites are particularly relevant in biomedical applications, such as implants, where strong adhesion to titanium surfaces is critical for long-term success. In summary, analyzing the adhesive forces, Young's modulus, and surface stiffness of HA-CS-composites provides insights into their potential for effective integration with titanium alloys, essential for various applications in the biomedical field [23, 24]. Because of these apparent improvements, this study intends to describe the next steps towards increasing the corrosion protection and surface properties of titanium alloy substrates that will be coated with HAP/CS/MgO layers utilizing the EPD technique. The study aims to investigate the influence of varying durations of electrophoretic deposition (EPD) on the surface roughness and thickness parameters of medical devices. These parameters play a critical role in determining the performance and durability of the devices. Furthermore, the research will delve into how these different EPD durations impact the corrosion resistance of titanium alloys, providing valuable insights for the design and manufacturing of medical implants and instruments. Taking into consideration the shortcomings of corrosion and low bioactivity of titanium alloys, this study attempts to modify the EPD conditions aiming at obtaining rather uniform and stable bioactive coatings, which greatly enhance the working period of medical implants.

2. Experimental procedure

2.1 Materials and Suspension preparation

The present research is composed of two parts. The titanium alloys Ti-6Al-4V were prepared for electrophoretic deposition (EPD) in the first part. The materials used in forming the coating EPD solution consisted of Hydroxyapatite (4 g/L), Chitosan (0.5 g/L) and Magnesium oxide (1.5 g/L). Substrate samples were cut into rectangular shapes of 15 × 15 × 3 mm dimensions. Thereafter, the standard samples were subjected to water cleaning followed by polishing with SiC papers having grits 400, 500 and 600 microns in size, stained with a 50:50 methanol/distilled water solution and allowed to dry in air. Thereafter, the Nano powders that were made up of suspension materials were dissolved in ethanol, citric acid and distilled water. After an agitation period of 30 minutes with a magnetic stir, an ultrasonic bath was used to avoid agglomeration. Table 1 gives a description of the preparation procedures and concentrations of the suspension material used to prepare the samples.

The second stage of the experiment included differences in the time values against the working Ti-6Al-4V, including 5V for the electrophoretic deposition (EPD) process for 10 minutes, which has already been coated with a compound of hydroxyapatite, chitosan and magnesium oxide.

The experimental procedure outlined in this work has been meticulously detailed to provide clarity and enhance understanding as shown in Figure 1.

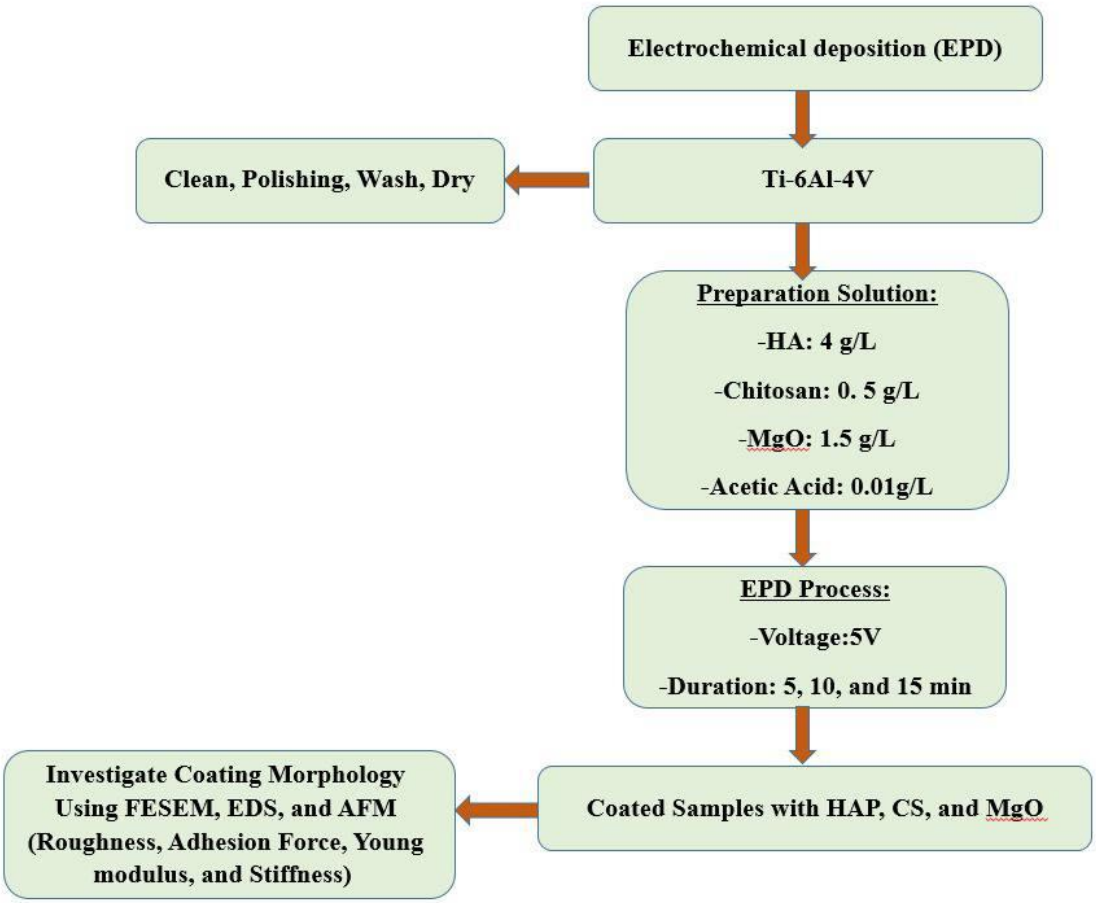

Figure 1. Schematic representation of the Electrophoretic Deposition (EPD) process.

Table 1. Suspension composition and process parameters of the EPD process.

No	Voltage (v)	Time (min)	CH (g/L)	MgO (g/L)	HAP (g/L)	No	Voltage (v)	Time (min)	CH (g/L)	MgO (g/L)
1	5	5	0.5	1.5	4	**1**	5	5	0.5	1.5
2	5	10	0.5	1.5	4	**2**	5	10	0.5	1.5
3	5	15	0.5	1.5	4	**3**	5	15	0.5	1.5

2.2 Characterization of the coating

Several tests were conducted to characterize the coating layer, the first important measure was Zeta potential, which is considered the main criterion to assess the stability of the suspensions and the homogeneity of the coatings. The study used the Zeta Plus Signal Processing utilizes Electrophoretic Light Scattering (ELS) to achieve a precision of ±3%, which may vary depending on the salt concentration which is 35 g/L. The system is equipped with a standard 35mW red diode laser with a nominal wavelength of 640nm. Field-emission Scanning Electron Microscopy (FESEM) technique and Energy-Dispersive X-ray spectroscopy (EDS) analysis (Inspect F 50 FEISEM, Eindhoven, The Netherlands) were used to investigate the thickness of the coating, surface morphology and element characterization of the coating on the surface of Ti-6Al-4V. The surface roughness, adhesion force, and mechanical performance (young modulus and stiffness) of the coating were evaluated using the Core AFM 2023 from Nano Surface AG, Switzerland.

3. Results and discussion

3.1 Zeta potential and particle size analysis

The study measured consistent Zeta potential values for all suspensions using the same concentration, dispersion methods, temperature, pH, and ionic strength. Multiple readings were taken before each measurement. Results are presented in Table 2, including particle sizes from the suppliers' data Material.

3.2 Thickness analysis of coating on the surface of Ti alloy

The across-sectional in Figure 2 shows the thickness of HA-CS-MgO on a Ti alloy deposited via electrophoretic deposition (EPD) at 5V for different times. The coating thickness experiences a substantial increase with extended deposition time. It starts at $17.1\pm0.84\mu m$ after 5 minutes, escalates to $36.5\pm0.7\mu m$ after 10 minutes, and further rises to $113.6\pm2\mu m$ after 15 minutes. The correlation between deposition time and coating thickness consistently demonstrates a trend of more than doubling in thickness as time progresses. Specifically, at 15 minutes, the thickness is nearly three times greater than at 5 minutes. The uniformity of the layers, especially in cases A and C, demonstrated a consistent structure devoid of any voids. The surface roughness analyzed through AFM images, along with the detailed SEM images of all samples—discussed in the following section—provides further confirmation of these observations. Remarkably, both cases exhibited no signs of voids or cracks, indicating a high-quality layer formation. The uniformity of these layers. across the substrate indicates that each deposition was effective at every time interval, resulting in thicker or denser coatings as EPD progresses for more periods. The correlation between deposition time and coating thickness is obvious from the fact that the latter increases more than twice as much when time increases. The homogeneity of these films over the substrate shows that each deposition was efficient at all-time intervals, hence leading to coatings becoming thicker or denser as EPD goes on for extended periods. The rise in coating thickness arises from the mechanism of electrophoretic deposition with longer times of EPD. At short deposition times, not many particles could reach the surface leading to a thin film. As the time of deposition increases, more particles are deposited and therefore form a thicker layer. The process speeds up because the initial coating improves how well surfaces capture other particles.

Moreover, with prolonged deposition time, one might expect that there is a compounded effect where increased area of coating growth and probably diminished repulsion between incoming particles and already deposited layer contribute to fast accumulation of material which brings about a larger increase in thickness. Therefore, the link between longer EPD times and increasing coat thicknesses can be explained by such sustained frequent EPD episodes over a longer period of time resulting in denser coatings than those formed within shorter periods.

Table 2. Particle sizes and suspension properties.

Materials	**Particle size**	**Suspension Zeta Potential (mV)**	**Suspension particles mobility (μ/s)/(V/cm)**
CH	Molecular Weight (g/mol) 1526.464	-39.8	-078
MgO	85% < 18 nm		
HAP	98.5%, <40nm		

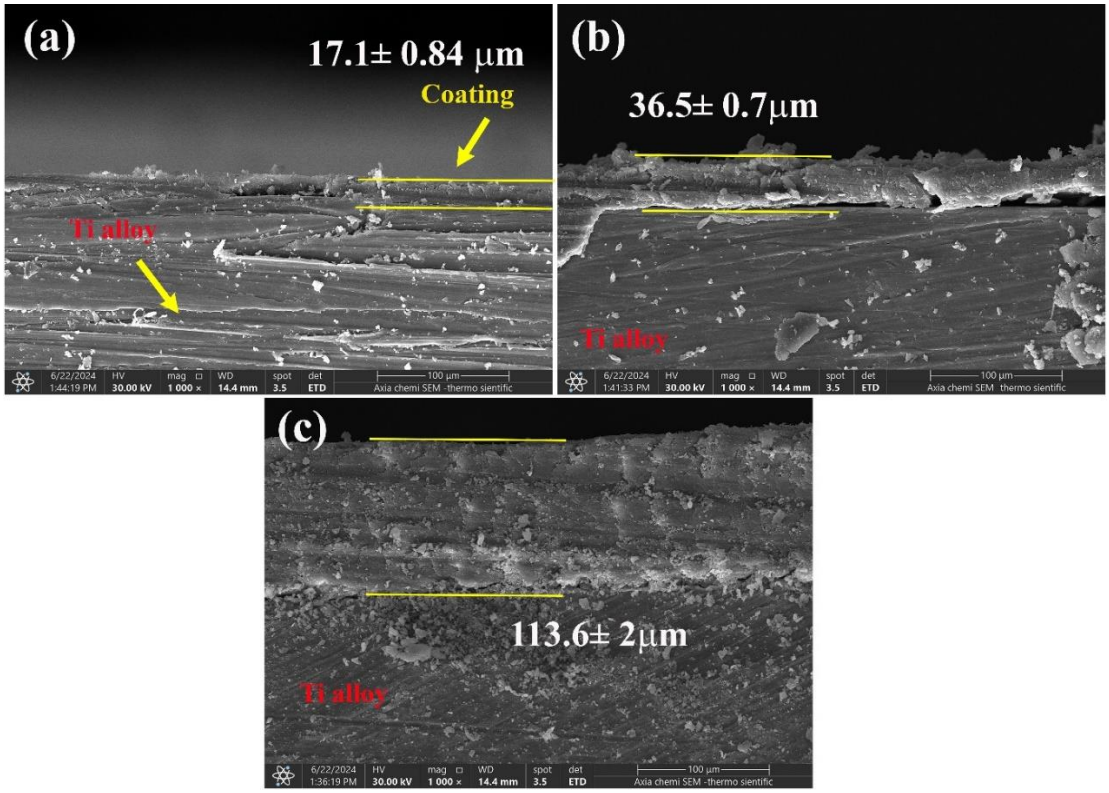

Figure 2. SEM Cross-sectional images showing the thickness of the Hydroxyapatite-Chitosan-Magnesium Oxide (HA-CS-MgO) composite coating on a titanium alloy substrate at different. coating time. a.5 min, b. 10 min, c. 15 min. The coating was deposited via electrophoretic deposition (EPD) at 5V for varying deposition times.

SEM images in Figure 3 present surface morphologies of the deposited HA-CS-MgO composite coating on a Ti alloy substrate via electrophoretic deposition at 5V for different duration times. The base metal (Ti alloy) has a polished smooth surface with visible smoothing scratches that characterize a well-prepared substrate for coating. Five minutes after the coating process begins, gaps are visible as a result of missing some part of the material, which shows an incomplete deposition. This means that the deposition is at its beginning stages and only a little material has been deposited so far to cover the surface entirely. As deposition time increases to 10 minutes, the coating becomes more uniform and continuous, reducing the presence of gaps and improving overall surface coverage. This shows that at longer deposition times, more particles are pushed towards the substrate resulting in better surface coverage and more uniform coatings. However, after fifteen minutes of deposition, while the coating completely covers the surface, cracks start to form probably because of internal stresses during its deposition or curing. Stresses of this kind may arise from the coating material shrinking as it dries or due to a mismatch in thermal expansion between coating and substrate. Cracks can even compromise the mechanical strength of a film and its protection, especially in applications requiring durability and environmental resistance.

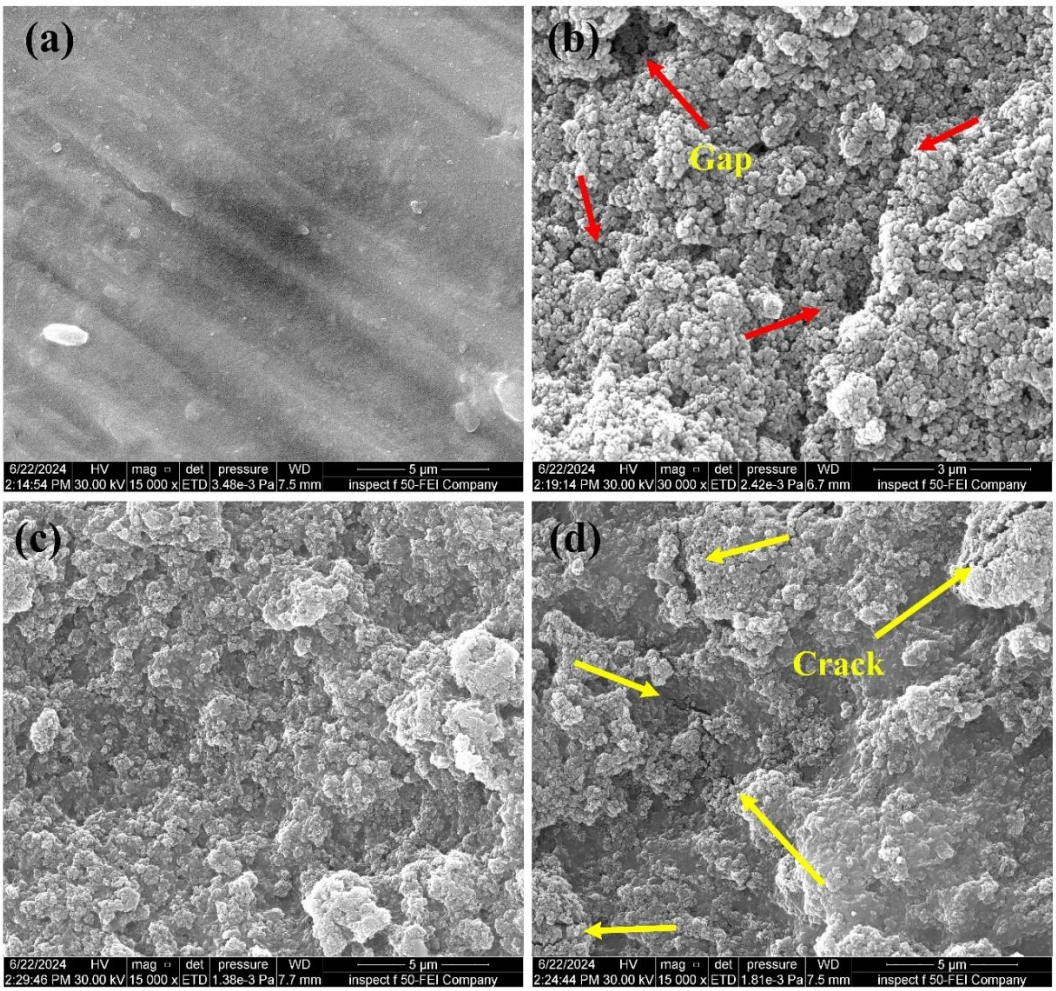

Figure 3. SEM top-sectional images showing the morphology of the Hydroxyapatite-Chitosan-Magnesium Oxide (HA-CS-MgO) composite coating on a titanium alloy substrate at different. coating time. a .base metal, b.5 min, c. 10 min, d. 15 min. The coating was deposited via electrophoretic deposition (EPD) at 5V for varying deposition times.

In the EDX analysis of a Ti-6Al-4V alloy before and after hydroxyapatite-chitosan-magnesium oxide coatings were applied via electrophoretic deposition (EPD) over different time intervals, distinct changes in elemental composition were observed Figure 4. The received base metal (Figure 4a) consists mostly of titanium 53.0 % but also contains considerable carbon and nitrogen and lesser aluminium and vanadium which rids the description from Ti-6Al-4V alloy. After a 5-minute coating (Figure 4b), there is a notable introduction of high levels of oxygen and calcium, characteristic of hydroxyapatite, alongside increases in carbon and the appearance of phosphorus, aligning with the hydroxyapatite-chitosan composite; trace elements like magnesium and silicon are also detected, likely from the magnesium oxide component. Extending the coating to 10 minutes (Figure 4c), the high percentages of calcium and oxygen suggest a more substantial coating, with titanium becoming undetectable, indicating complete coverage by the coating materials and a slight reduction in carbon while maintaining significant phosphorus levels. By 15 minutes (Figure 4d), the coating shows even higher levels of calcium and oxygen and an increase in phosphorus, indicating a denser and more integrated coating with a slight rise in carbon content, possibly due to the increased presence of chitosan. These progressive changes underline the EPD's effectiveness in modifying the surface properties of the alloy for enhanced biomedical applications.

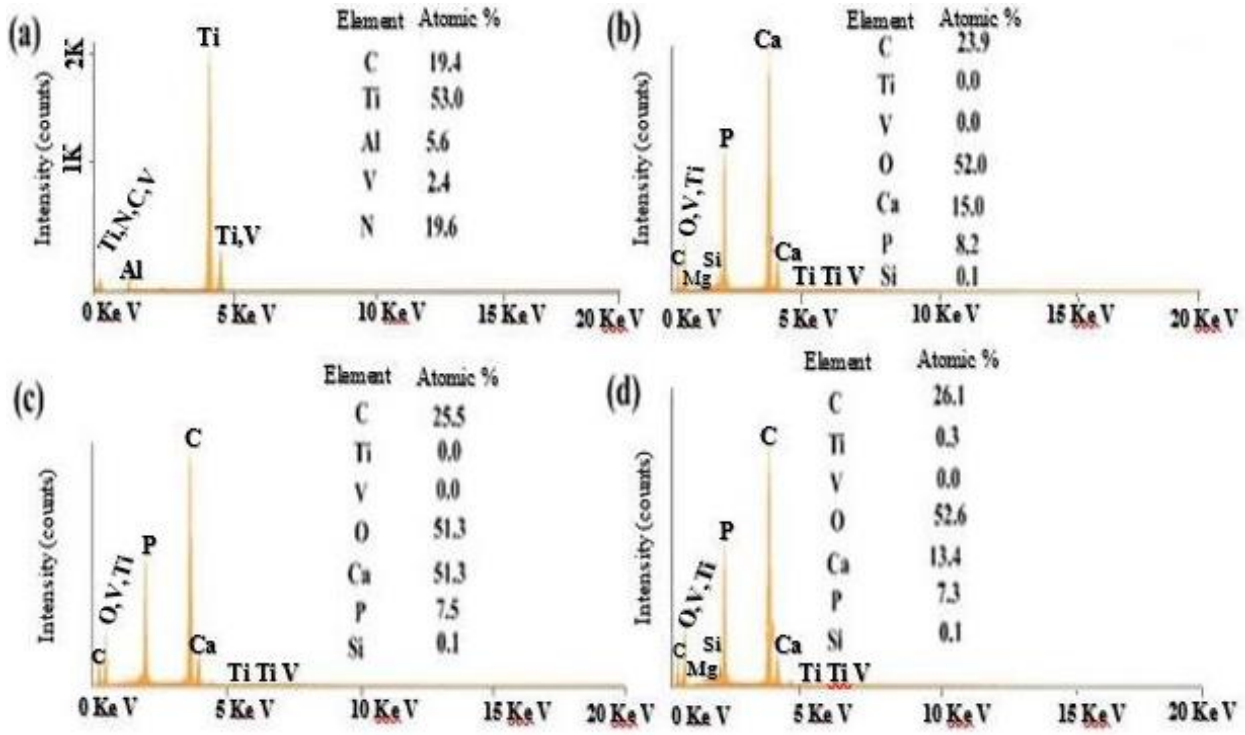

Figure 4. The elemental composition and Energy Dispersive X-ray Spectroscopy (EDX) spectra of Ti-6Al-4V alloy were examined both before and after electrophoretic deposition of coatings consisting of hydroxyapatite, chitosan, and magnesium oxide, with different durations applied. (a) base metal, uncoated. (b) Coat for 5 min, (c) for 10 min, (d) for 15 min.

3.3 AFM analysis surface of Ti alloy coated by HA-CS-MgO

The results of the AFM analysis are presented in Figure 5 and Table 3, which demonstrate how the roughness of HA-CS-MgO composites on a Titanium alloy substrate change over time during electrophoretic deposition. The AFM analysis results are presented in Figure 5 and Table 3, illustrating the evolution of the roughness of HA-CS-MgO composites on a Titanium alloy substrate during electrophoretic deposition. . The uncoated Ti alloy surface initially was very rough with high values of Sq (361.7 nm) and Sa (311.3 nm), which indicates that its surface had prominent ridges and valleys. After 5 minutes of deposition, the surface becomes much smoother as seen from the reduced roughness (Sq = 136.5 nm, Sa = 103.9 nm). It implies that initial coating smoothed out surface irregularities to produce an even and smooth surface. For 10-minute coatings, this downward trend in roughness persists (Sq = 93.8 nm, Sa = 77.9 nm), reflecting a more evened coating with improved coverage reducing imperfections on the external interface throughout the metal surface. However, after 15 minutes of deposition, the surface roughness increases again (Sq = 248.7 nm, Sa = 212.6 nm). An AFM image indicates that this increase in roughness may have resulted from the generation of bigger clumps, aggregations, and maybe defects such as cracks which interrupt the earlier smooth surface. Such an increase in roughness upon extended deposition time demonstrates that although initial deposition enhances surface smoothness, prolonged deposition times might result in the development of undesired surface characteristics. This emphasizes the need for optimizing the length of deposition time to strike a balance between getting a smooth uniform coating and avoiding over-deposition-induced degradation of surfaces.

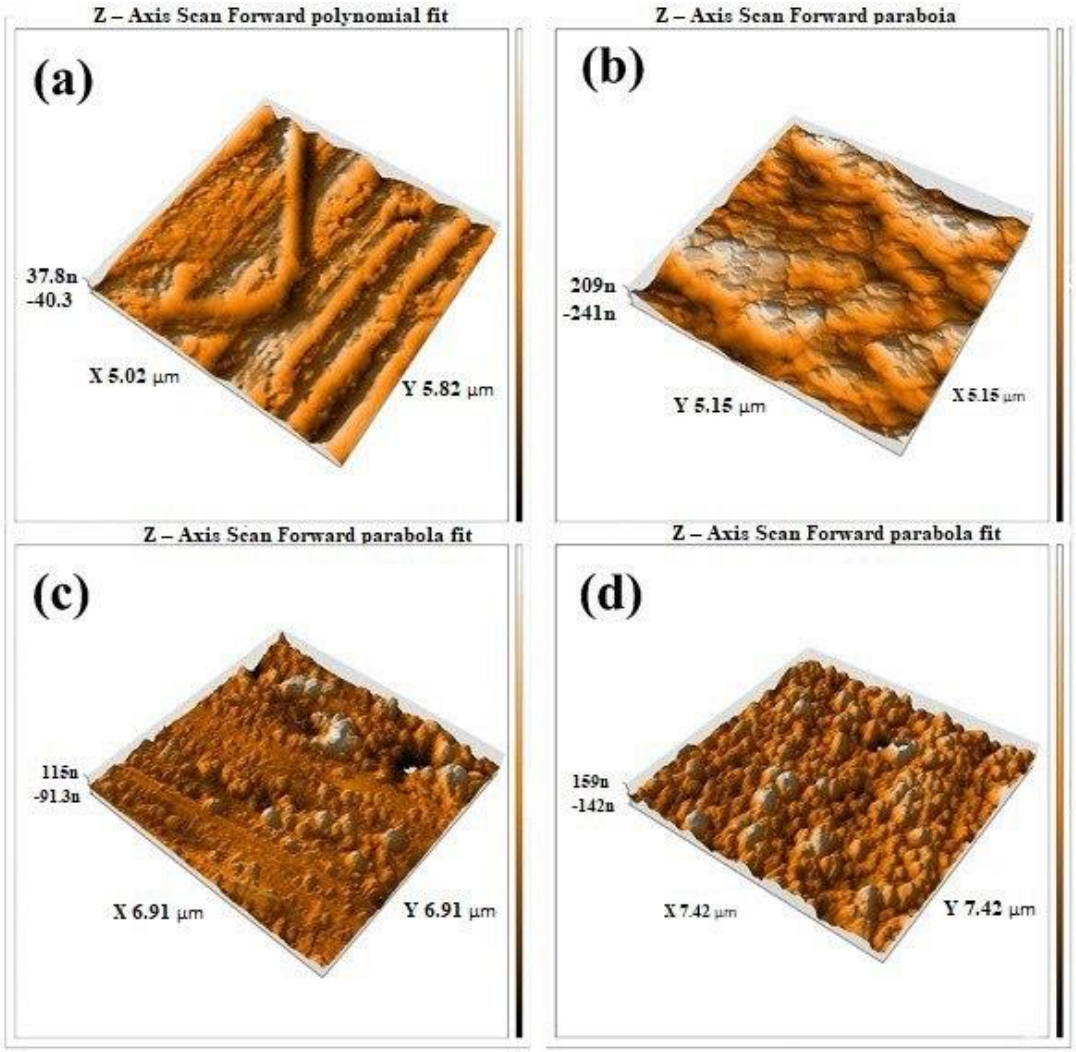

Figure 5. Atomic Force Microscopy (AFM) images illustrating the changes in surface roughness of Hydroxyapatite-Chitosan-Magnesium Oxide (HA-CS-MgO) composites on a titanium alloy substrate over time during electrophoretic deposition at different. coating time. abase metal, b.5 min, c. 10 min, d. 15 min.

Table 3. Surface roughness of HA-CS-MgO coatings on titanium surfaces at different coating time

.

Sample	Duration of coating (min)	Roughness	
		(Sq) nm	(Sa) nm
a	**Without coating**	361.7	311.3
b	5	136.5	103.9
c	10	93.8	77.9
d	15	248.7	212.6

3.4 Mechanical performance analysis of the coating on the Ti alloy surface

The analysis of the adhesive force is illustrated in Figure 6. Table 4 shows, shows Young's modulus and surface stiffness of the hydroxyapatite-chitosan-magnesium oxide (HA-CS-MgO) composites deposited on the titanium alloy surface by electrophoretic deposition. These properties are significantly dependent on the coating duration. The best compromise was achieved with a 10-minute duration which produced the highest adhesion (-1.36 nN), Young's modulus (9.69 MPa) and

surface stiffness (0.34 nN/nm); thus forming a well-adhered, stiff and durable coating. As such, ten minutes appear to be a sufficient time for fabricating compact well-structured coatings with strong adhesion to the substrate. In contrast, a 5-minute coating duration results in moderate adhesion force (-0.39 nN), Young's modulus (7.37 MPa), and surface stiffness (0.29 nN/nm). While the data suggests that the coating maintains a relatively stable structure, the shorter time frame may not provide adequate conditions for the coating to fully form. This phenomenon can lead to properties that are only partially developed, creating mechanical weaknesses in comparison to the ten-minute sample. The latter exhibits significantly stronger and more reliable characteristics. Notably, these observations align with the findings reported by Li-Chong Xu [22] and colleagues, further supporting the validity of this analysis.

Moreover, an additional decline in performance is observed when the duration is extended from 10 to 15 minutes. During this period, the adhesion force shifts to a slightly positive value of 0.05 nN, indicating a minimal attachment rather than a strong bond. Furthermore, Young's modulus experiences a notable reduction to 3.93 MPa, reflecting a decrease in the material's stiffness, while surface stiffness diminishes to 0.19 nN/nm, indicating a loss of rigidity across the coating's surface. These changes suggest that extended timeframes could compromise the overall integrity and functionality of the coating. The length of deposition suggests that hard coating is formed with thicker and more porous ones that are not tightly adherent to the substrate thereby decreasing their overall mechanical strength. It is therefore advisable to use a 10-minute deposition time as this will ensure the best combination of mechanical properties and good adhesion in the coating

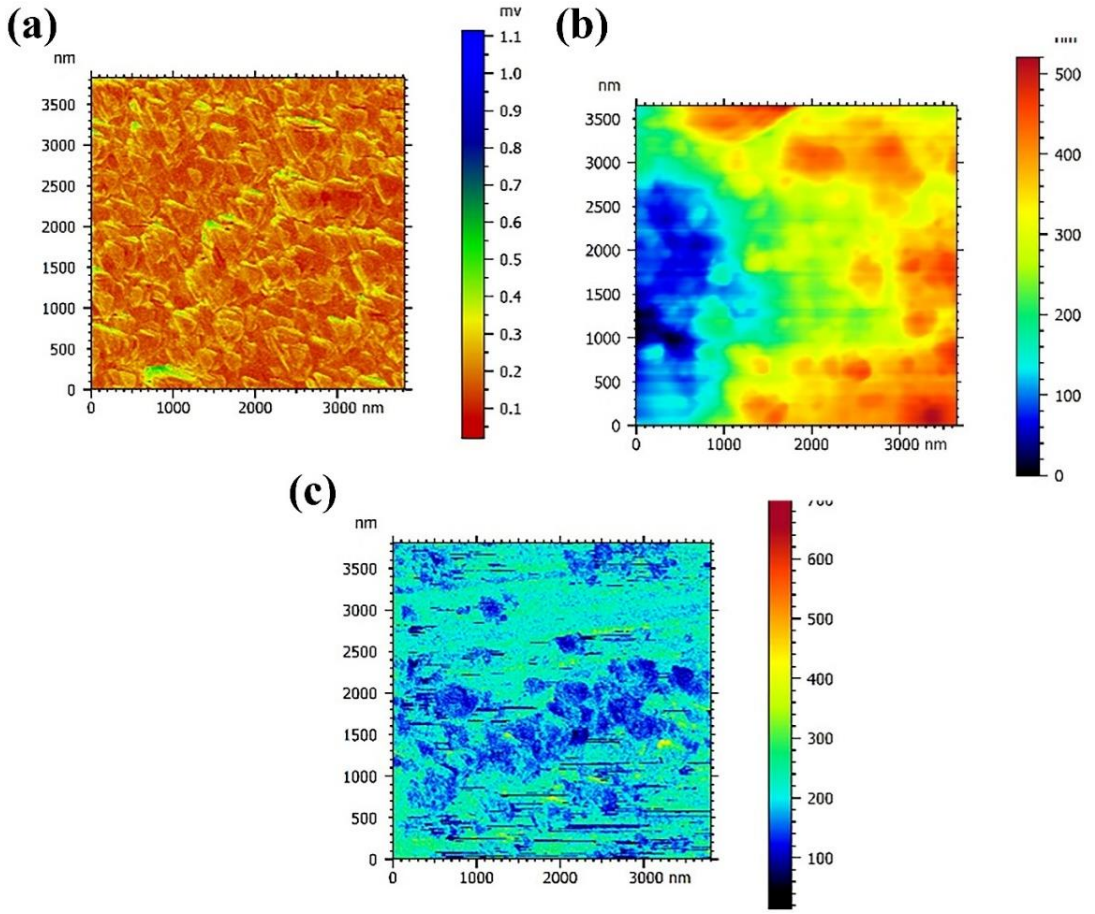

Figure 6. Presents atomic force microscopy (AFM) analysis showcasing adhesion map images of (HAP-CS-MgO) composites coated on a Ti alloy substrate. These images were obtained using electrophoretic deposition (EPD) at 5V for different durations: (a) 5 minutes, (b) 10 minutes, and (c) 15 minutes.

Table 4. Surface roughness of HA-CS-MgO coatings on titanium surface at different coating time.

Sample	Duration of coating (min)	Coating properties		
		Adhesion force (nN)	Young's modulus (MPa)	Surface stiffness (nN/nm)
a	5	-0.39	7.37	0.29
b	10	-1.36	9.69	0.34
c	15	0.05	3.93	0.19

4. Conclusion

The research work presented here was able to show for the first time the effects of the deposition time on the characteristics and properties of Hydroxyapatite-chitosan-magnesium oxide (HA-CS-MgO) composite coatings on the titanium alloy Ti-6Al-4V using electrophoretic deposition (EPD) at 5V, with coating durations ranging from 5 to 15 minutes. The results indicate the following:

1. As the deposition time increased (5-10min), factors such as the coating thickness, surface morphology, roughness and adhesion force, Young modulus, and stiffness of the coatings were improved. The 10-minute deposition time proved to be the most appropriate with a coating that was uniform, dense and smooth with the best mechanical properties including the highest adhesion, Young's modulus and surface stiffness.
2. Increasing the coating time to 10 minutes increased calcium and oxygen levels, indicating a more robust coating. Additionally, titanium became no longer detectable, indicating that the coating material completely covered the titanium. Carbon content also decreased slightly, while phosphorus content still increased significantly.
3. The study observed that while a longer duration of coating deposition leads to increased thickness, it does not necessarily enhance performance and may introduce undesirable cracks and defects. Consequently, a well-bonded and mechanically sound HA-CS-MgO coating on Ti-6Al-4V alloy can be attained with an optimal electrophoretic deposition (EPD) time of 10 minutes.
4. The research findings indicate that the adhesion force transitions to a marginally positive value of 0.05 nN, suggesting a weak attachment rather than a robust bond. Additionally, Young's modulus shows a significant decline to 3.93 MPa, which signifies a reduction in the stiffness of the material. Concurrently, the surface stiffness decreases to 0.19 nN/nm, highlighting a loss of rigidity on the surface of the coating during the period from 10 to 15 minutes.
5. The current work suggested a further investigation is needed to optimize the length of deposition time to strike a balance between getting a smooth uniform coating and avoiding over-deposition-induced degradation of surfaces.
6. Future work is suggested to fully cover the other characteristics such as contact angle, anti-bacterial test, and biocompatibility.

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