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Effect of Applied Voltage and Coating Time on Nano Hydroxyapatite Coating on Titanium Alloy Ti6Al4V Using Electrophoretic Deposition for Orthopaedic Implant Application

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Abstract. Nanoparticles of Hydroxyapatite (HA) were coated onto biocompatible titanium alloy, Ti6Al4V ELI using Electrophoretic Deposition (EPD) in order to improve its bioactivity. Electrophoretic Deposition (EPD) has been selected as the coating method because of the simplicity of the instrument, inexpensive cost, and ability to coat complicated products. This study, therefore, aims to investigate the effect of voltage and coating time of EPD process on increasing of implant mass as a parameter of deposition rate, coating thickness, and surface coverage of the HA on implant screw prototype products. Voltages were controlled in the range of 3, 5, and 7 volt and coating times were in the range of 3, 5, and 7 minutes. Surface morphology was examined using scanning electron microscopy (SEM). Coating thickness was measured by coating thickness gauges. While surface coverage was determined using ImageJ software. Based on the result, applied voltage and coating time affects the mass growth of samples and HA coating thickness in positive correlation. However, on the surface coverage, applied voltage and coating time reach the optimum value at 5 volt and 5 minutes. The best HA coating in which fulfilling the standard for orthopaedic implants was obtained at 5 volts for 5 minutes with mass growth is 0.00107 g, coating thickness are 79.13 µm, and surface coverage is 97.89%. HA coating thickness that produced in this research has fulfilled the desired coating thickness for orthopaedic implant application (50-100µm). SEM micrographs show that nano-HA is coated the alloy surface uniformly at these parameters. It can be concluded that these parameters can be applied to coat titanium Ti6Al4V ELI with HA for improving bioactivity in the orthopaedic application.

Keywords: Hydroxyapatite, Electrophoretic Deposition, Ti6AL4V ELI, Orthopedic Implant

1. Introduction

Nanoparticles of Hydroxyapatite (HA) are coated onto biocompatible titanium alloy, Ti6Al4V ELI using Electrophoretic Deposition (EPD) in order to improve its bioactivity in orthopaedic implant application[1]–[3]. Hydroxyapatite (HA) had been used worldwide for coating material on a metallic implant for orthopaedic application in order to accelerate bone fracture healing[4]–[8]. The increasing cases of metallic implant failure made the hydroxyapatite coating become one of the good solutions for the problems such as aseptic loosening, metal ion release that can be the trigger metal allergy, and chronic inflammation[9]–[11]. Electrophoretic Deposition (EPD) has chosen as coating method because of the simplicity of the instrument and its making, inexpensive cost, and ability to coat things with the complicated design[12]–[16]. EPD used electric current to move the HA particle through electrode in the suspension of ethanol and HA[14], [17], [18]. Desired HA coating quality can be adjusted by optimizing the voltage and coating time[19]–[21].

Ti6Al4V ELI is the titanium alloy that designed to use for orthopaedic implant application. This material has good mechanical properties such as desired strength for load bearing and dynamic load. However, the inertness of titanium made the Ti6Al4V ELI still have no bioactivity, so that, it needs to be coat with HA for gaining bioactivity properties[22]–[26]. This research aimed to analyze the effect of voltage and coating time of EPD process toward HA coating that produced on the surface of titanium alloy, Ti6Al4V.

2. Materials and Methods

2.1 Sample Preparation

Ti6Al4V ELI Titanium bar in length of 400 mm and 4 mm in diameter was cut and lathe into screw shape with type M3 x 0.5. Screw length and diameter were 5 mm and 3 mm, respectively. Then, the screws were sandblasted to refine the screw surface and clean the remaining waste from the lathe process. After that, screws were cleaned using ultrasonic cleaner in 15 minutes.

2.2 Coating Process using Electrophoretic Deposition

Pretreatment was conducted before the coating process. Screws were submerged in ethanol, acetone, and HNO3 in 15 minutes, respectively. After that, screws were remaining in NaOH for about one hour. HA suspension was made of 1.8g HA powder and 50 ml ethanol, and pH was set about 4. Nano HA powder that used was the commercial Nano HA. The anode was graphite and cathode was Ti6Al4V titanium. Voltages were in the range of 3, 5, and 7 volts and coating time were in the range of 3, 5, and 7 minutes. After the coating process screws were sintered in a vacuum furnace at 700oC with holding time about one hour. This method was modified from the research that has been conducted before[14], [16], [18].

2.3 Hydroxyapatite (HA) coating Characterization

Characterization of HA coating was conducted by an optical microscope that directly connected with a digital camera to obtain the microstructure images of HA coating and continued to assess surface coverage value using software ImageJ. In addition to, screws mass growth was also assessed to confirm the deposition of HA on the screws surface. After that, HA coating thickness was assessed using coating thickness gauges.

3. Results and Discussion

3.1 Surface Morphology of HA coating on the screw surface

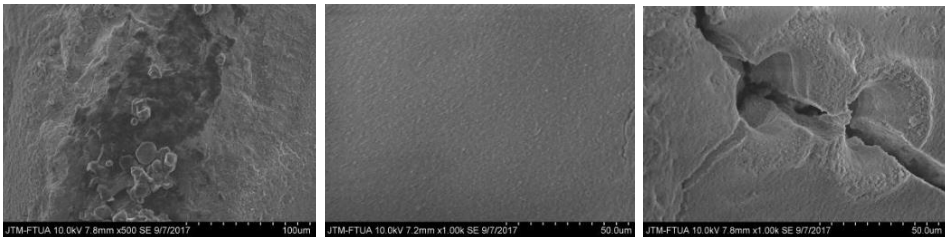
Figure 1 shows the HA coating morphology that captured by an optical microscope for each voltages treatment. Based on the image it is shown that there are significant differences on the surface morphology of each sample. The enhancement of voltage will produce HA coating that spread evenly but also has optimum value. Sample from 5-volt treatment shows the best surface coverage of HA coating especially at the thread of screw region. Thread of screw is the important part of the screw that directly contacted with the bone tissue after the implantation, so it is necessary to sure that this region has been coated very well with no empty space that possible the corrosion after long time application.

3 volts 5 volts 7 volts

Figure 1. Optical Microscope imaging of surface morphology of HA coating on the screw surface at different voltage

However, the sample from 7-volt treatment shows cracks on the surface of the HA coating. It can be assumed that HA coating that produced by 7-volt treatment has a high thickness that made the bonding between HA particle and titanium surface become weak and lead to cracking. It is also mean that voltage applied higher than 5 volts would produce HA coating with low quality, in other words, there is an optimum value of the applied voltage that should be considered to produce HA coating on Ti6Al4V ELI using EPD.

A research that conducted previously also revealed the same result with the current research. Increasing of the applied voltage from 5V to 20V will produce HA coating that spread more evenly and thicker. However, applying high voltage on EPD process will produce crack on the coating layer that indicated the high voltage is not suitable to produce good HA coating layer on the Ti6Al4V ELI implant surface[17]. Using high voltage up to 200 volts seems to be not recommended to produce desirable HA coating for orthopaedic implant application because the difficulties to produce the crack-free coating that would be impacted the performance of the implants[2].



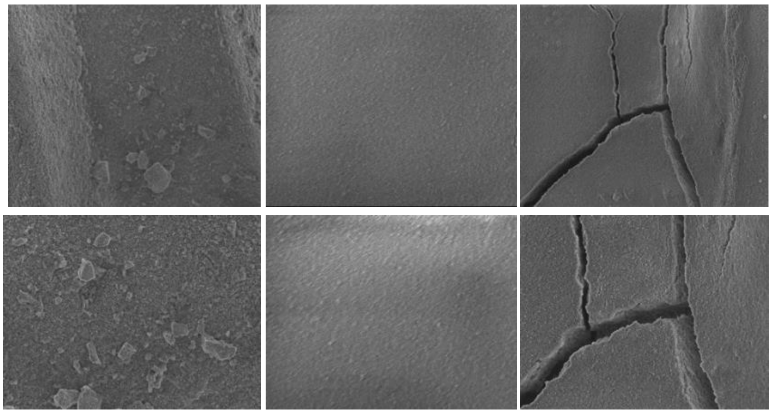
3 volts 5 volts 7 volts

Figure 2. SEM imaging of surface morphology of HA coating on screw surface at different voltage

SEM imaging (fig.2) also shows the same result with optical microscope images. Sample from 5-volt treatment shows the best surface coverage and there are no cracks can be found. It can be assumed that sample from 5-volt treatment can be sample candidate to produce orthopaedic implant using EPD process. Optical Microscope imaging of surface morphology of HA on the screw surface at different coating time (Fig. 3) showing a similar result with the voltage treatment (Fig. 1). Sample from 5 minutes shows the best result because the HA powder spread more evenly and cracks are not found especially at the thread of screws region. The SEM imaging of the samples also shows the same result (Fig. 4). Another research also revealed the same result with this current research. The deposition of HA particle will increase in compliance with the increasing of coating time. However, it seems to be there is an optimum point of coating time that will produce a better HA coating layer[12], [16], [27]. Based on this result it can be assumed that using coating time up to 7 minutes will produce HA coating that will have the crack on its surface.

3 minutes 5 minutes 7 minutes

Figure 3. Optical Microscope imaging of surface morphology of HA coating on the screw surface at different coating time



3 minutes 5 minutes 7 minutes

Figure 4. SEM imaging of surface morphology of HA coating on screw surface at different coating time

3.2 Mass Growth of Specimen

Table 1. Mass Growth of Specimen at Different Voltage

No.	Voltage	M_1 (g)	M_2 (g)	ΔM (g)	Average Δm (g)
1	3	0.1848	0.1852	0.0004	0.000333
2		0.1965	0.1968	0.0003	
3		0.2120	0.2123	0.0003	
1	5	0.2372	0.2380	0.0008	0.001067
2		0.2182	0.2192	0.0010	
3		0.2100	0.2114	0.0014	
1	7	0.2212	0.2223	0.0011	0.001567
2		0.2139	0.2153	0.0014	
3		0.2307	0.2329	0.0022	

Table 1 shows the mass growth of specimen after EPD process at a different voltage. This result shows the same pattern with the imaging result from optical microscope and SEM. Based on this result it can be seen that the enhancement of applied voltage will increase the mass of the screws. This data are collected to confirm the existence of HA particle that adheres on the surface of Ti6Al4V screws. Enhancement of screws mass will show the deposition of HA particle on the screws surface. Based on the data, it can be seen that the specimen from 7-volt treatment has the highest mass growth. It means that the enhancement of voltage will increase the HA particle deposition. It can be caused by the enhancement of potential differences on the cathode and anode that produce high energy to throws HA particle onto screws surface that causes higher HA particle deposition. Figure 5 shows the graphic of mass growth of screws at a different voltage in average. Another research also revealed the same result with this current research, increasing the applied voltage will increase the deposition of HA particles significantly[16], [27].

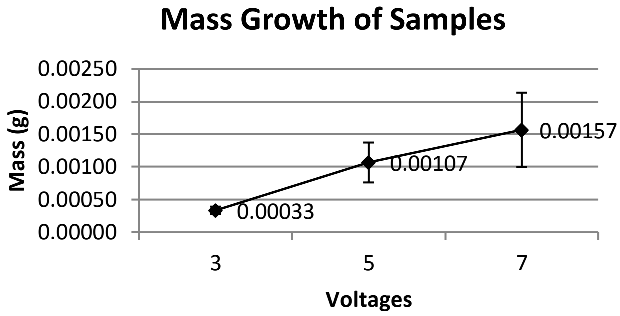


Figure 5. Average of screw's mass growth at different voltage

In compliance with the mass growth of specimen from voltage treatment, the mass growth from the specimen for coating time treatment also revealed a significant result. Table 2 shows the mass growth of specimen after EPD process at different coating time. Based on the data, it can be seen that the enhancement of coating time will increase the mass of the screws. The highest mass growth is found at the specimen from 7 minutes treatment. It can be concluded that enhancement of coating time will increase the deposition of HA particle on the screws surface. Another research also revealed the same result with this current research, increasing the applied voltage will increase the deposition of HA particles significantly[12], [16], [27]. Figure 6 shows the graphic of mass growth of screws at different coating time in Figure 6 shows the graphic of mass growth of screws at different coating time on average. However, this data has not shown the optimum value yet. So that, it is necessary to continue the investigation to understanding the effect of voltage and coating time of the EPD process on the Ti6Al4V implant that could produce optimum HA coating for better orthopaedic implants.

Table 2. Mass Growth of Specimen at Different Coating Time

No.	Time (minutes)	M_1(g)	M_2(g)	ΔM(g)	Average Δm (g)
1	3	0.2002	0.02005	0.0003	0.00043
2		0.2226	0.2231	0.0005	
3		0.2519	0.2524	0.0005	
4	5	0.2372	0.238	0.0008	0.00093
5		0.21	0.2114	0.0014	
6		0.2182	0.2188	0.0006	
7	7	0.1991	0.2009	0.0018	0.0014
8		0.2376	0.2394	0.0018	
9		0.2236	0.2244	0.0008	

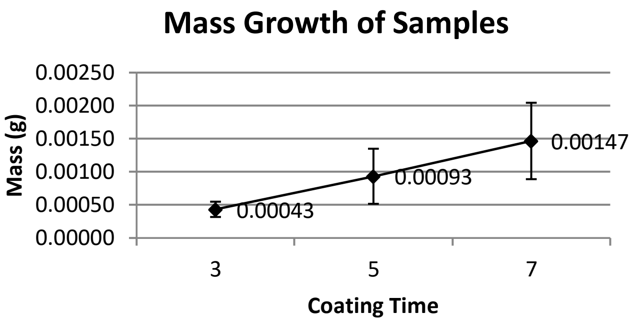


Figure 6. Average of screw's mass growth at different coating time

3.3 Coating Thickness

Table 3. HA Coating Thickness that produce at different voltage

No.	Voltage	Thickness (µm)	Average (µm)
1	3	57.6	54.23
2		54.4	
3		50.7	
1	5	81.2	79.13
2		79.1	
3		77.1	
1	7	90.9	92.13
2		93.1	
3		92.4	

Table 3 shows the data of HA coating thickness that produce on the surface of Ti6Al4V ELI screws implant. Based on the data, it can be seen that the enhancement of applied voltage on the EPD process will increase the HA coating thickness that produces on the surface of the screws. The highest coating thickness is 93.1 µm found at the 7-volt specimen and lowest coating thickness is 50.7 µm found at the 3-volt specimen. However, all the thickness data have fulfilled the characteristic for the orthopaedic implant application. The optimums HA coating thickness that suitable for orthopaedic implant application are between 50-100 µm[28]–[30]. But, the coating thickness is not the only parameter that should be considered in evaluating the ability of an orthopaedic implant since the surface coverage, chemical composition on the implant surface, hydrophobic feature, etc must be considered[28].

In addition to, the effect of HA coating thickness on the titanium implant surface must be concerned for the next research. Because the HA coating thickness on the titanium implant will affect the ability of an implant to keep the biocompatibility and bioactivity features. Except, HA coating thickness also affect the corrosion resistant and shear strength of the metal implant as the result of the reaction that influenced by the biological environment of the human body[31], [32]. Figure 7 shows the HA coating thickness that produces at a different voltage in average. Based on the graphic it can be concluded that enhancement of applied voltage will increase the coating thickness.

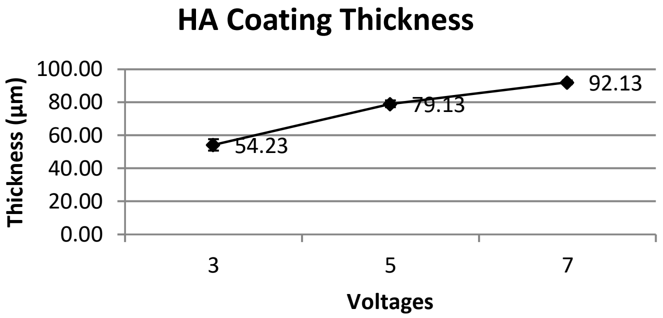


Figure 7. Average of HA coating thickness that produce at different voltage

HA coating thickness that produces on the surface of Ti6Al4V ELI screws implant at different coating time also showing the same pattern with the specimen from voltage treatment. Enhancement of coating time will increase the HA coating thickness on the Ti6Al4V ELI screws implant surface. The highest coating thickness is 94.9 μm found at 7 minutes specimen and the lowest coating thickness is 58.62 μm found at 5 minutes specimen (Table 4 and Fig. 8.).

Table 4. HA coating thickness that produce at different coating time

No.	Time (minutes)	Thickness (μm)	Average (μm)
1	3	66.66	63.94
2		66.52	
3		58.62	
1	5	77.56	78.16
2		79.8	
3		77.14	
1	7	94.9	90.38
2		87.8	
3		88.44	

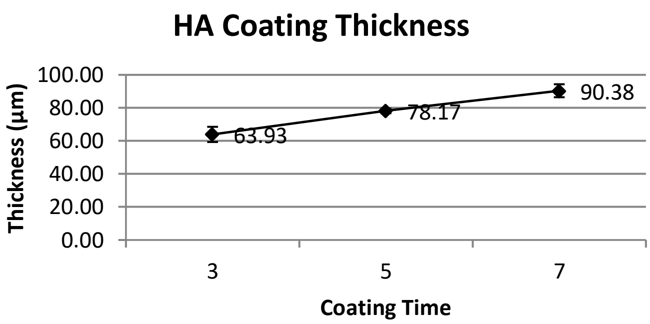


Figure 8. Average of HA coating thickness that produce at different coating time

3.4 Surface Coverage

Figure 9 shows the surface coverage data of HA coating on the surface of Ti6Al4V ELI screws implant. Based on the data it can be seen that the highest surface coverage is about 97% found at the 5-volt specimen and the lowest surface coverage is 73% found at the 3-volt specimen. However, at the 7-volt specimen the surface coverage value is decreased. It can be concluded that there is an optimum value of the applied voltage that can produce better HA coating on the Ti6Al4V ELI screws implant. Surface coverage is an important parameter for evaluating the quality of HA coating on the implant. Data from coating time treatment also shows the same pattern with the voltage treatment. The best surface coverage is about 82% found at 5 minutes specimen (Fig. 10.). Another research also revealed that HA coating that prepared at a constant low voltage consisted of fine HA particle and was dense. While the HA coating that prepared at constant high voltage consisted of big HA particle and was porous[15].

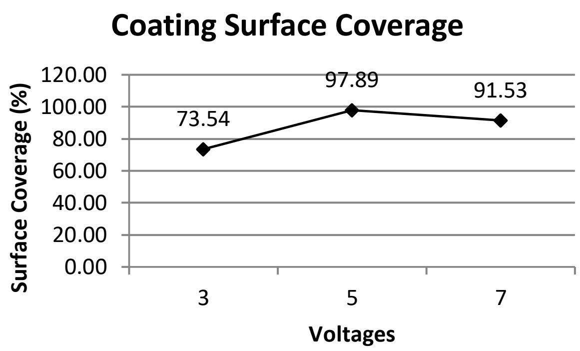


Figure 9. Surface Coverage of HA coating at different voltage

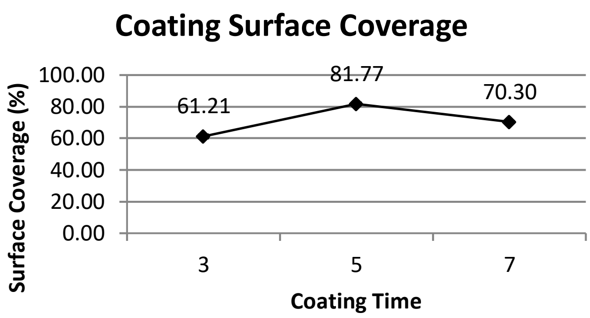


Figure 10. Surface Coverage of HA coating at different coating time

4. Conclusions

1. Applied voltage and coating time affects the mass growth of samples and HA coating thickness in positive correlation.
2. However, on the surface coverage, applied voltage and coating time reach the optimum value at 5 volt and 5 minutes.
3. The best HA coating in which fulfilling the standard for orthopaedic implants was obtained at 5 volts for 5 minutes with mass growth is 0.00107 g, coating thickness are 79.13 µm, and surface coverage is 97.89%.
4. HA coating thickness that produced in this research has fulfilled the desired coating thickness for orthopaedic implant application (50-100µm).

5. Acknowledgements

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