A Study the Effect of porosity of Bio-Active Ceramic Hydroxyapatite Coated by Electrophoretic Deposition on the Ti6Al4V Alloy Substrate

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Abstract. Bone implant materials which are composed of the Ti-6Al-4V alloy are usually utilized as a result of its bio-compatibility and corrosion resistance. This alloy isn’t bio-active, and that results in making it easy to release the cation Al and V when implanted into the human body. Therefore, this study aims to focus on the effect of different current direct current (DC) and alternating current (AC) on the electrophoretic deposition (EPD) variables (voltage, time and concentration) of the Hydroxyapatite (HA) powder upon the Ti6Al4V alloy substrate. EPD process was depended on the coating thickness and surface roughness, and then characterizations of the coating layer were performed. The suspensions zeta potential was measured to recognize charge upon the particles and their stability. The coating thickness was measured at different EPD variables. The cross-section of coating thickness and the morphology of surface were investigated employing optical and SEM microscopes. The density of coating porosity was computed from the micrograph images. Results manifested that the samples having the same thickness possess a higher grain density if coated utilizing (AC) EPD in comparison to (DC) electrophoretic deposition. The contact angles values of coatings have been changed from hydrophobic in the case uncoated Ti6Al4V 67.489°C to the hydrophilic by using (DC) current 35.914°C and 38.13 °C by using(AC).

Keywords: EPD, AC and DC, Hydroxyapatite (HA) surface roughness, Ti6Al4V alloy, biomedical coatings

1. Introduction

Presently, the artificial biomedical prosthesis in addition to the standard surgical-grade material implants, like stainless steel (316L), Cobalt-Chromium (Co-Cr) alloys and Alloys of Titanium (Ti) are widely utilized in the dentistry and orthopedics field. Bio-medical devices and their components, such as senses, organs, dental, orthopedic, screws and bone plates are used as implantable materials for aiding, healing or correcting abnormalities of the systems of affected ones due to its unique mechanical and
biological properties [1]. Alloys of Titanium (Ti) are amongst the most broadly utilized metallic implants in the orthopedic and dental uses due to their biocompatibility, high strength to weight ratio, and exceptional resistance to corrosion [2]. Due to their fast passivation nature, the better capability of osteo-integration in the biological regime in comparison with the other implant materials is due to many attractive physicals, mechanical and biological properties. As well, Titanium and its alloys are broadly utilized in short-term and lasting fixation instruments, like bone screws and plates, Tibial constituents of Total Knee Arthroplasty (TKA) [1]. The perfect implants should have improved adhesion to the tissue of human alongside with antibacterial activity, bioactivity, and enhanced resistance to corrosion for preventing the failures of implant owing to the shortage of bioactivity and the bacterial infection [3]. Such characteristics could be enhanced via either the modifications, like the wettability and the surface roughness or via the bioactive and antibacterial coatings deposition [4]. Ti and Ti alloys, such as Ti-6Al-4V, are identified due to their superior tensile strength as well as the resistance of pitting corrosion [5]. EPD is extensively utilized as a cost-effective and easy method for a diversity of coating uses [6,7]. The other technologically employed calcium phosphate coating methods comprise, plasma spraying deposition, electrostatic spray deposition and pulsed laser deposition [8, 9]. However, direct current-electrophoretic deposition (DC ) and alternating current-electrophoretic depositions (AC) are approaches where the subjected voltage has been provided from a (Direct current) field or an (Alternating current) field, correspondingly [10]. For direct current-electrophoretic deposition method, the charged particles deposition takes place in suspension to the reversely charged electrode, beneath the fixed electric field effect. In alternating current-electrophoretic deposition method, the electric field direction is periodically inverted [11]. That is responsible for the oscillation and migration of the particles of powder within suspension among the electrodes. That oscillating migration is reliant upon the asymmetry and frequency of the applied wave in the electrophoretic deposition process [12] and [13]. Hydroxyapatite) HA was widely utilized in the medical and dental requests due to its near chemical composition with normal bone tissues, in addition to its high bio-compatibility; none-the-less, the pure HAP is suffering from the weak mechanical characteristics [14] and [15]. Contrary to the HAP, the Ti6Al4V alloy has been confirmed of being a suitable material for the load-bearing requests. Which has resulted in the concept of the Ti-6Al-4V alloy coating with the HAP combines the advantages of the two substances [16] and [17]. The time and hydroxyapatite (HA) powder concentration upon Ti6Al4V plate were conducted utilizing ethanol as solvent beneath the direct current (DC), (EPD) variables (voltage and alternating current (AC) fields depending on the roughness and the thickness) and then characterize the coating layer [18] and [19]. The suspension stability and the particles dispersed charge are generally two significant measures for the electrophoretic deposition (EPD) [20] and [21]. This study aims to clarify the study of the difference between alternating current and direct current to precipitation bioactive ceramic on the substrate of Ti6Al4V alloy with ethanol. To optimize the electrophoresis process (EPD) using a different parameter (voltage, time and concentration). Hydroxyapatite is a biologically compatible phase and ethanol provides sufficient stability of hydroxyapatite in the suspension Through the electrophoresis process. XRD analysis and SEM observations were used for characterization purposes. Micrographs were analyzed for estimating the density of surface porosity and density of deposition (depending upon the
percentage of pore area of cross-section). The likely mechanism of coating for (Hydroxyapatite) in the two methods was debated from the surface and cross-section morphology.

2. Experimental Work and Procedure

The Ti-6Al-4V plates having 5 mm thickness were first to cut into samples by wire cut machine, and the dimensions were 10 X 20 mm from the alloy substrate, which has been utilized as working electrode (cathode), while the stainless steel (316L) was used as an anode. These substrate samples were polished using 400, 600 and 800 grits of silicon carbide papers. Then, they were first washed by ethanol, then via acetone and finally by water having a high purity inside an ultrasonic bath for (20 min) high-energy sonicator (Ultra-sonic Processor, MIXSONIX INC, NY, US). Beyond the cleaning process operation, the plates have been draying until being used later.

EPD suspension has been produced through the use of 3 concentration levels 1, 2 & 3g/L Hydroxyapatite (40nm, 99% purity, and white color with 3. 14g.cm⁻³ density) which has been purchased from (Sigma Aldrich) which is used to deposit it as layers of coating. Chitosan (medium molecular weight, which has a de-acetylation level of approximately (85%) soluble in (1%) acetic acid which has a 98% purity), which has been purchased from Sigma Aldrich, has been utilized as a binder on the Ti-6Al-4V and with the HAP powders. The significant step in EPD experimental process is preparing aqueous suspensions. The initial step has been the dissolution of 0.50g/L of the chitosan with the use of 1.0% of the acetic acid [19]. Then, the purity of the solvent ethanol (has been higher than 98%) added with 2.5.0% of the distilled water and then HAP material Nano-powder has been added. All of the suspensions have been de-agglomerated through magnetic stirring prior depositing to be homogenized then stirred with the use of a magnet at a 200rpm speed. It has been followed with a high-energy sonicator for 30min and a 24h magnetic stirring. A (pH) meter in addition to the acetic acid has been used to the modification range of (pH=4) of the solutions with the use of the (pH-EC-TDS Meter Portugal). The Zeta potential has been evaluated for every one of the solutions, for the purpose of ensuring its stability. EPD cell which has been utilized in this research includes a beaker and 2 electrodes that have been immersed in suspension as the schematic EPD system which has been utilized. The distance still fixed at (1.0cm) between electrodes. Fig1 depicts EPD system which has been utilized in this research.

![Electrophoretic Deposition system EPD](image)

**Figure 1.** Electrophoretic Deposition system EPD
The material which has been utilized in this study, has been prepared as plate, was the Ti6Al4V which has been tested in Science and Technology Ministry through the use of the analytical instruments (model XEPOS which is referred to as SPECTRO). Table 1 lists the (Ti-6Al-4V) alloy’s chemical composition.

Table 1. Chemical composition analysis of the Ti-6Al-4V alloy

| Chemical analysis | Al  | C  | Fe  | V   | N   | H   | O   | Ti   |
|-------------------|-----|----|-----|-----|-----|-----|-----|------|
| Ti6Al4V (Standard)| 5.5-| 0.08| 0.3 | 3.5-| Max | Max | Max | Balance |
| (Measured value)  | 5.94| 0.007| 0.25| 4.04| 0.021| 0.0018| 0.073| Balance |

The (AC) signal utilized was a square kind having an asymmetry factor (3) reached throughout a programmable function generator. The signal frequency was (1000 Hz). Table 2 shows the samples description and the used parameters of deposition.

Table 2. Deposition voltages and times utilized in the current study with the respective sample symbols

| Method Process                                      | Voltage (V) | Time (sec) | Sample Symbol |
|-----------------------------------------------------|-------------|------------|---------------|
| Direct current-electrophoretic deposition            | 20          | 240        | DC-1          |
|                                                     | 40          | 240        | DC-2          |
|                                                     | 60          | 240        | DC-3          |
| Alternating current-electrophoretic deposition       | 20          | 360        | AC-1          |
|                                                     | 40          | 360        | AC-2          |
|                                                     | 60          | 360        | AC-3          |

The measured the suspension’s pH value, which has been equal to 4, led to obtaining the zeta potential with a positive value as has been listed in Table 3 and Fig. 2 which enhances the homogeneity of the HA precipitation. The increase in the mobility is through the increase in the absolute values of zeta potential. The high mobility of HA particles returns to the positive value of the zeta potential [2]. The measurements of zeta potential were conducted employing a zeta probe (DT1200) (Dispersion Technologies Inc.) with (3g/L) HA suspensions arranged in pure ethanol and preserved beneath the conditions of stirring. The zeta potential and mobility HA NPs have been illustrated for suspension, as can be seen in Table 4 and Fig 2.

Table 3. Results of the HAP Mobility and Zeta Potential

| Material | Frequency | Frequency Shaft | Mobility | Zeta Potential |
|----------|-----------|-----------------|----------|---------------|
| 3-1 HA   | 2A8.09    | -1.91           | 0.25     | 21.74         |
This research focused chiefly upon the techniques of deposition for assessing the influence of the applied fields (DC or AC) upon the behavior of the density as well as porosity percentage inside the deposited layer. The surface of the chosen samples was characterized by the X-ray diffractometer; the coated samples cross-sections were arranged for the thickness measurements of coating. The morphology of surface and the coated samples cross sections were performed utilizing the Field Emission Scanning Electron Microscope (VEGA3LM TESCAN COMPANY). The percentage of surface porosity area and the percentage of pore area alongside the coatings’ thickness have been computed employing the (J-Image) software, Version (1.46) [20]. The computed results from the (3) micrographs were averaged. To calculate the percentage of the average pore area utilizing the J-Image, a rectangular area was chosen upon the (Hydroxyapatit) zone of the chosen micrographs. The value of threshold was modified for best fitting through the area calculation of the particle. Here, pores have been regarded as particles, and the resulted percentage of area has been computed from the area of pores, which were detected within choosing. The Ti6Al4V alloy and the stainless steel 316-L have been respectively cathode and anode. The procedure of the deposition has been taken place through a variety of the conditions of the deposition at 1, 2 and 3g/L concentrations, times of HAP 2, 4 and 6min, and 20, 40 and 60 V voltage values. Next, at the room temperature, the samples coating has been dried with air to 24 hours.

3- Results and Discussion
The HA-Ethanol suspension zeta potential was (21.74 ± 0.40mV) (Mean ± Standard deviation). The suspension has revealed no particle sedimentation signs (via the visual observation) for a minimum of (24 hour). The (XRD) for a single layer of Hydroxyapatite deposited on the alloy substrate showed the presence of different diffraction peaks as compared with powder of HA. This can be attributed to the chitosan particles, which act as a binder material and as a coating thin layer on Ti6Al4V substrate which affected the XRD peaks. Fig. 3 illustrates the phases of HAP layer (002), (211), (300), (222), (213 and (004) . The highest intensity for HA was found at 2Ø 32.25º. Figure 3 (a) also depicts the XRD analyses for Ti-6Al-4V alloy in as-received conditions. It has explained the fact that structure has been consisting of (α+β). The α phase represents the α-stabilizing element as Al element tends for the stabilization of α phase, whereas the (V) element acts as a β phase stabilizer [23].

![Figure 2. Results of Zeta Potential of (HA)](image)
4- Measurement of the Contact Angle

The hydrophobicity or the wettability of coated Ti-6Al-4V alloy and the uncoated samples have been measured with the use of a steady distilled water drop through the measurement of solid and liquid contact angle through the optical contact angle equipment of the CAM 110-04 W type that has been attached with the CCD camera coating decreases the angle of the contact Ti-6Al-4V substrate from 67.489º to 38.132 º in the case of the coating with the (AC) and 35.914º with the use of the (DC) that in turn turns uncoated Ti-6Al-4V alloy surface from hydrophobic to hydrophilic. The coating of the Surface has been more hydrophilic in a layer of the HAP coating. In such case bone regeneration becomes more rapt and easy. When there are decreases in the contact angle with the HAP coating has been deposited on the coating of the substrate. Which has given results that have been in agreement with [23]. The above description of the values of the contact angle for the uncoated and coated Ti-7Al-4V alloy have been illustrated in Figure 4.

Figure 4. Water contact angle measurements of (a) Uncoated Ti6Al4, (b) HAP- coated Ti samples by using(AC) and.(C) Contact Angle of HAP with (DC) current.

5-Analysis of SEM

5.1. Electrophoretic deposition by using direct current (DC)

For producing samples with raising the thickness of the coating, the voltage was raised from (20 to 60 V). The deposition time was (240 sec) in the whole cases, Table 2. This deposition initiated merely from (20 volts) which could be owing to the comparatively lower particles electrophoretic movement. Fig. 5 represents the development of surface (a, b, d, e, g, and h) and cross-section (c, f, and i) morphologies of the direct current-electrophoretic deposition coatings via the increase of voltage. The cracking was noticed inside the whole non sintered samples [22]. The micrographs cross-section showed that the coating close to the substrate of (Ti6Al4V) consists of particles, in a smaller size, that are well packed, resulting in a thicker coating. Faraway from the interface, the coating is developed via the bigger particles of (HA) and it converts further porous
Figure 5: Micrographs of SEM of the surface and cross-section morphologies of various (Direct current-electrophoretic deposition) coating

The scale bars are 100 μm for Figure 5 (a, d, and g) and 10 μm for Figure 5 (b, c, e, f, h, and i), correspondingly. The micrographs of the cross-section elucidated that the thickness of coating rose with the increment in the exerted voltage. Such values of thickness are listed in the Table 2. The least used voltage was (20 V) for the deposition that caused within the (HAP) coating thickness (23 μm). The voltage increase to (40 V) caused increase thickness to more than (23 μm). The computation of the percentage of porosity was achieved upon the surface micrographs for understanding the development of the density of the surface porosity. The outcomes are evinced in the Table3 These values manifest that the percentages of porosity that noticed upon the (Direct current-electrophoretic deposition) samples are alike regardless of the change in the parameters of deposition, this result agreement with reference [23].

Table 4. Parameters of deposition, thicknesses and the computed densities of porosity of the (DC-EPD) samples

| Name of sample | Voltage (V) | Coating Time (sec) | Average Thickness (μm) | Total Area (μm) | Average Size (μm) |
|----------------|-------------|--------------------|------------------------|------------------|-------------------|
| DC-EPD-1       |             |                    |                        |                  |                   |
| DC-EPD-2       |             |                    |                        |                  |                   |
| DC-EPD-3       |             |                    |                        |                  |                   |
5.2. Electrophoretic Deposition By Using Alternating Current (Ac)

To produce samples with uniform coating thickness of those direct samples coated with electrostatic deposition, different combinations of voltage time were used. Table 3 illustrates these parameters. Accurate SEM diagrams of the coatings arranged via the AC-EPD method are depicted in Figure 5. Surface morphologies show distinct pore behavior compared to current direct electrophoresis samples. To switch between the current electrophoresis, the pores are lower compared to any other samples in the present study. However, greater pores were observed with increased voltages and coating thickness.

|       | Voltage (V) | Time (sec) | Thickness (nm) | Porosity (%) |
|-------|-------------|------------|----------------|--------------|
| DC-1  | 20          | 240        | 23              | 21973        | 7.312        |
| DC-2  | 40          | 240        | 25              | 42900        | 12.570       |
| DC-3  | 60          | 240        | 26              | 37911        | 15.324       |

Figure 6. SEM micrographs of the morphologies of surface and cross-section of various (Alternating current-electrophoretic deposition) coatings.

The scale bars are 100 μm for Figure 6 (a, d and g) and 10 μm for Figure 6 (b, c, e, f, h and i), respectively. The values of thickness are given in Table 4. An increment exists in the thickness of the coating beyond a (60 V) active voltage increment (AC-EPD-2) or a (360 sec) time (Alternating current-electrophoretic deposition -3). Also, the porosity percentages are listed in Table 5 as in the (Direct current-
electrophoretic deposition) case, the parameters deposition in the (Alternating current-electrophoretic deposition) didn’t vary the density of crack in a considerable way.

**Table 5.** Parameters of deposition, thicknesses and the computed densities of porosity of (AC-EPD) samples.

| Name of sample | Voltage (V) | Coating time(sec) | Average thickness(μm) | Total Area (μm) | Average Size(μm) |
|----------------|-------------|-------------------|-----------------------|-----------------|-----------------|
| AC-1           | 20          | 360               | 24                    | 77970           | 2.372           |
| AC-2           | 40          | 360               | 28                    | 14842           | 1.943           |
| AC-3           | 60          | 360               | 31                    | 39357           | 2.921           |

The results of density of porosity exhibited in the Tables 3 and 4 manifest that the surface porosity percentage area in the (Alternating current-electrophoretic deposition) samples case is (20-28%) lower than the (Direct current-electrophoretic deposition) samples. Regardless of the conditions of deposition, the porosities percentage was same as that within the utilized method.

5.3. Porosity Test

The percentage of porosity in (HA coating) was measured utilizing the image analysis on the micrographs of the cross-section for comparing discrepancy to the (Alternating current-electrophoretic deposition) as well as (DC-EPD) coated samples, and the samples having the same thickness of coating (DC-EPD-1 and in alternating current-electrophoretic deposition-3) were chosen. The used technique was same as the one explained earlier for the surface determination of porosity. The computed porosity was (20%) and (11%) for (DC-EPD-1) and (In alternating current-electrophoretic deposition-3), respectively. Therefore, the alternating current-electrophoretic deposition coated sample was thicker indicating a better mechanism of packing in the (AC-EPD) technique case. For understanding the mechanism of deposition, samples with the same coating-thickness (Alternating current-electrophoretic deposition-3, Direct current-electrophoretic deposition-1) were chosen. Micrographs of coating layer elucidated further thickly dense coating to the Alternating current-electrophoretic deposition-3

![Figure 7](image-url) The comparison of the cross-sections of coating gained between (AC) and DC-EPD

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At Fig. 7 Evinces a cross-sectional coating layer (Alternating current-electrophoretic deposition -3) that indicates a thickly packed coating composed mainly of small and medium particles. The other side found more particles of greater size in the case of the (direct current-electrophoretic deposition -1) coating sheet, as shown in Fig. 6. The particles are treated with an oscillation according to the signal used (as shown in Fig. 6a) in (alternating current-electrophoretic deposition). The specific wave results in a migration of the (Ti6Al4V) particles [6]. Hence the significance of asymmetry in the case of alternating current-electrophoretic deposition is important. The probability of migration of the smaller grain is higher at comparatively high frequencies, and hence better governance of the grain size needed for coating is possible. Co-deposition of bioactive HAP has also been seen, mainly through applications for DC and AC-EPD. For orthopedic and bone tissue engineering applications, HAP-chitosan coatings are ideal because they will result in superior bonding between the implant and the bones around it. Such coatings are also degradable at different speeds, making them useful candidates for releasing local drugs or therapeutic ions to investigate the electrophoretic co-deposition of specific current (AC and DC) HAP chitosan composite coatings on Ti6Al4V substratum. For the development of this type of coatings, AC-EPD was used to minimize bubble formation and film disruption during the deposition process; this finding is in line with the reference [24].

6. Conclusions

The following are the summarized concluded points of the present work:

1. The results manifested that the (AC EPD) technique gave a dense coating and less porosity than (DC-EPD) for the same thickness.
2. (AC-EPD) is useful for sediment powders with wide distribution of nanoparticle size, and this method can also control particle transmission according to wave inconsistency and frequency.
3. Tuning the (AC) wave asymmetry, and the frequency, can more enhance the characteristics of the coating that will be involved in the next investigation for the present study.
4. Zeta potential test confirmed the stability of solution for depositing the coating and it is (21.74) for 100% HA + 0.5% Chitosan.

7. References

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