Facile Coating of HAP on Ti6Al4V for Osseointegration

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Abstract—Ti6Al4V alloy is a material with great strength, low-slung modulus, inferior density, and a virtuous blend of mechanical and exceptional corrosion resistance. However, it does not offer good osseointegration and bone development properties. Conversely, hydroxyapatite (HAP) is highly bioactive in nature to bind with the nearby bone tissues when implanted in the host body. In this work, we have extracted HAP from bovine bones by using the thermal decomposition method. This was followed by its deposition onto the Ti6Al4V alloy using the Electrophoretic Deposition (EPD) technique. TiO$_2$ is used as a bond coat layer to increase the adhesion between HAP and Ti6Al4V alloy substrates. The coated samples after sintering exhibited excellent adhesion. This was followed by characterization using Scanning Electron Microscopy (SEM) and Fourier Transformed Infrared Spectroscopy (FTIR). FTIR and SEM confirm the formation of HAP and its presence after the immersion in SBF. Vicker hardness tester confirms the increase in hardness value of coated samples up to 35%. Potentiostat tests were conducted to compare the corrosion rate of both samples. In addition, the particle sizes were also identified by a laser particle analyzer, whereas X-Ray Diffraction (XRD) technique was also used to determine the crystalline phases of alloy and HAP.

Keywords—corrosion; electrophoretic deposition; hydroxyapatite; simulated body fluid; Ti6Al4V alloy

I. INTRODUCTION

Materials having biotic nature or host tissue compatibility can be implanted into living organisms to augment or replace impaired parts. Generally, metals, ceramics, polymers, and composites are materials widely used in biomedical applications. Amongst them, titanium-based alloys are some of the most widely used as implant materials [1]. Titanium and its alloys bear enhanced properties like excellent strength [2], optimum elastic modulus [3], low density [4], blend of other mechanical properties [5], and excellent corrosion resistance [6-12], needed for various applications including orthopedic [13], dental [14], and surgical implants [15], artificial joints [15], etc. It should be noted that the elastic modulus of titanium-based alloys is considerably close to bones’ [16, 17] which makes it ideal for long term applications [18]. However, titanium based alloys do not possess good osseointegration [19] thereby requiring additional surface treatment. This means that there is a poor bond between titanium and bones causing implant loosening which is highly undesirable. Therefore, surface modification plays a vital role in optimum osseointegration [15, 20]. There are several techniques used in this regard such as: sand blasting [21, 22], etching [23], electrochemical treatment [24], and thermal spray coatings [25]. Amongst them, bio-ceramic coatings using, the hydroxyapatite (HAP - Ca$_{10}$(PO$_4$)$_6$(OH)$_2$) [26-28] are very promising for the modification of implant surfaces since they create strong bonding with bones [29-31]. One of the great advantages of the HAP is its great lifespan [32, 33]. The calcium phosphate (CaP) ratio of HAP is 1.67 which is highly stable at a normal temperature and its pH ranges from 4 to 12. However, the properties and resultant applications of HAP depend on morphology, size, chemical composition and crystallinity [31]. In addition, it provides speedy and durable fixation to the host bones and possesses osseoconductive properties [34], protecting the metal surfaces from the environmental effects and thereby reducing the discharge of metallic ions from the implant surface to the host body. The main advantage of CaP is that it is already present in the bones and teeth of the vertebrates [35]. Moreover, there are several methods to produce HAP such as: dry methods, wet methods, microwave (MW)-assisted methods, ball-milling or ultrasound, etc. [36]. Additionally, numerous coating strategies are available to coat HAP on metallic alloys, for instance: plasma spraying [37, 38], sol gel [39, 40], Electrophoretic Deposition (EPD) [11, 41-43], etc. Amongst them the most economically viable technique is EPD [44], offering a controlled coating composition with the process being highly pure [45], fast [41]
that could be used to coat complex shaped substrates [45]. In this study, HAP was produced from bovine bones since they are an abundant and economical source. This was followed by the deposition of HAP on chemically treated Ti6Al4V alloy.

II. EXPERIMENTAL PART

A. Materials

The materials utilized in this research are shown in Table I. All of them were of analytical grade and were used in as received condition.

| S.No. | Chemicals / materials | Purity (%) | Supplier |
|-------|-----------------------|------------|----------|
| 1     | Ti6Al4V               | 99.0       | Baoji North Hongsheng Industry & Trade Co., Ltd. |
| 2     | Sodium chloride       | 99.0       | Sigma Aldrich |
| 3     | Sodium bicarbonate    | 99.0       | Sigma Aldrich |
| 4     | Potassium chloride    | 99.0       | Sigma Aldrich |
| 5     | Di sodium phosphate trihydrate | 99.0 | Sigma Aldrich |
| 6     | Magnesium chloride hexa hydrate | 99.0 | Sigma Aldrich |
| 7     | Hydrochloric acid     | 99.0       | Sigma Aldrich |
| 8     | Calcium chloride dehydrate | 99.0 | Sigma Aldrich |
| 9     | Di sodium sulphate    | 99.0       | Sigma Aldrich |
| 10    | Cyano hydride tri methanol | 99.0 | Sigma Aldrich |

B. Methods

1) Preparation of Hydroxyapatite

HAP was synthesized from bovine bones, purchased from the local market, by the thermal decomposition process. This was followed by their boiling in deionized water for about 3 hours to remove the unwanted fats. Additionally, second boiling was carried out for the deproteinization of bones. Thereafter, the bones were immersed in acetone for two hours in an ultrasonic bath for further cleaning. Subsequently, the bones were dried and cut into smaller sizes by mortar and pestle. The resultant powder was placed into a box furnace and heated at 1100°C at a heating rate of 5°C/min for 3 hours for the preparation of HAP. Later on, XRD and laser particle analysis were conducted to confirm the peaks and particle sizes of as-synthesized HAP powders respectively. The process details are shown in Figure 1.

3) EPD of HAP

For the enhancement of adhesion properties of hydroxyapatite, Titania (TiO$_2$) was used as an intermediate layer between HAP and Titanium alloy substrate. The process flow chart is shown in Figure 2. In this process, an electrolytic solution containing 0.5g TiO$_2$ and 0.5g HAP was dissolved in 100ml ethanol solution followed by sonication for 15 minutes and was ultrasonically shook for 30 minutes. It was then left for settling for 20 minutes and was stirred again for 25 minutes at 40°C. The EPD process was done at 20V for 5 minutes while keeping the distance between electrodes at 2cm. The resultant coating was sintered in the tube furnace at 800°C. Finally, the samples were ready for characterization to further evaluate the properties of Ti-6Al-4V alloy coated with hydroxyapatite.

**Fig. 1.** Steps of producing HAP from bovine bones, calcination cycle, and XRD pattern of successfully produced HAP.

**Fig. 2.** Process flowchart of coating HAP on Ti-6Al-4V substrate.
III. RESULTS AND DISCUSSION

A. Particle Size Analysis

We used the BT-9300H laser particle analyzer for the determination of particle size of HAP powder. The results are shown in Figure 3. The average obtained particle size was 25.73 microns with respect to cumulative percentage.

![Average particle size of HAP powder synthesized by bovine bones.](image)

**Fig. 3.** Average particle size of HAP powder synthesized by bovine bones.

B. XRD Analysis

The X-pert Pro XRD DY3313 XRD machine operating at 40kV and 30mA using CuKα radiation was used to analyze the phase of the HAP. The diffraction pattern was recorded over 20° to 79°. Figure 4 reveals the XRD spectra of bovine bones calcinated at 1100°C which is in good agreement with the standard HAP pattern [46, 47]. The calcinated sample peak at 31.8 confirms the formation of hydroxyapatite powder. Moreover, the XRD spectra of the chemically treated Ti alloy surface show the presence of TiO2 and coated alloy confirming the presence of the HAP layer on the chemically treated Ti6Al4V.

![XRD pattern of synthesised HAP, chemically treated surface and Titanium alloy coated with HAP powder after sintering.](image)

**Fig. 4.** XRD pattern of synthesised HAP, chemically treated surface and Titanium alloy coated with HAP powder after sintering.

C. Chemical Composition

The chemical composition of titanium alloy is found by using an X-ray fluorescence spectroscope (INNOV-X SYSTEMS) which is comparable to ASTM F136 standard [48]. The results are presented in Table II.

| Alloy      | Titanium | Vanadium | Aluminum |
|------------|----------|----------|----------|
| ASTM F136  | Balance  | 3.5-4.5  | 5.5-6.75 |
| Ti6Al4V    | Balance  | 4.17     | 6.12     |

**TABLE II. CHEMICAL COMPOSITION USING XRF ANALYSIS**

![Chemical Composition using XRF analysis](image)

D. Microstructures

The polarized light microscope OLYMPUS GX51 was used to reveal the microstructure of the as-received samples. It was found that the titanium alloy consists of two different phases: equiaxed Alpha and transformed Beta phase [49] which are shown in Figure 5(a). Figure 5(b) shows the SEM image of the HAP powder. The powder comprises of agglomerated fine particles while the shape of the particles is angular and non-spherical [50]. Figure 5(c) shows Ti6Al4V alloy coated with HAP after sintering which revealed that there was an enhanced linkage and the interconnection of HAP powders that exist on the coated surface morphology [51]. Figure 5(d) shows the HAP coated on Ti6Al4V after the immersion in SBF [52].

![Microstructures](image)

**Fig. 5.** (a) Optical structure of as received sample. (b) SEM image of the produced HAP powder. (c) Ti6Al4V coated with HAP and sintered at 800°C. (d) After immersion in SBF solution.

E. Hardness

Micro Vickers hardness tester (DIN EN 6507) was used to examine the hardness of bare and coated Ti6Al4V alloy by applying a load of 500g with a dwell time of 10s. The results are shown in Table III. A significant increase in hardness value of 468HV was obtained for a coated sample as compared to the pristine sample with a hardness value of 340HV. The Ti-based alloy produced a high hardness value which is a major requirement in being compatible with the body environment.

![Hardness results](image)

**TABLE III. HARDNESS RESULTS**

| Alloy            | Hardness (HV) |
|------------------|---------------|
| Ti6Al4V (before coating) | 340           |
| Ti6Al4V (after coating)  | 468           |
F. Corrosion Behavior

1) Immersion Test of Coated Samples in SBF

The coated samples of the HAP were immersed in Simulated Body Fluid (SBF) solution which was prepared according to the procedure defined in [40]. The chemical composition of the SBF solution is shown in Table IV. The characterization was based upon the pH scale. For comparative analysis, the pH of the solution was measured before immersion and was found to be 7.4. The sample was then immersed in the solution enclosed in a glass beaker at 37°C for 7 days. Agitations and vibrations were provided to the beaker to create an artificial environment of fluid movement around the coating. After a passage of the prescribed time, the sample was removed from the solution. The solution pH was measured after the immersion and was found to be the same as before, i.e. 7.4. Therefore it was confirmed that no exchange of ions took place between the sample and SBF.

| S.No. | Chemicals                     | Amount     |
|-------|-------------------------------|------------|
| 1     | Sodium chloride               | 6.559g     |
| 2     | Sodium bicarbonate            | 2.26g      |
| 3     | Potassium chloride            | 0.3773g    |
| 4     | Di potassium phosphate tri hydrate | 0.1496g |
| 5     | Magnesium chloride hexa hydrate | 0.3411g |
| 6     | Hydrochloric acid             | 10ml       |
| 7     | Calcium chloride dehydrate    | 0.3635g    |
| 8     | Di sodium sulphate            | 0.0731g    |
| 9     | Cyano hydride tri methanol    | 6.0662g    |

2) FTIR

FTIR (Perkin Elmer spectrum one system) was used to identify the functional groups of HAP coated samples in the area of 400-4000cm⁻¹. The FTIR spectra before and after the immersion in SBF solution is shown in Figures 6 and 7. The PO₄³⁻ group displays peaks at 560 and 600cm⁻¹ and at 1000–1100cm⁻¹. The peak at about 2600–3600cm⁻¹ links to the hydrated OH⁻ ion of HA [53]. The peak from 2000 to 2200cm⁻¹ shows the stretching of P-O-H [54]. The peak at 1490cm⁻¹ matches CO₃²⁻ which specifies that HAP is a carbonated-apatite (HCA) [55]. The representative peaks of PO₄³⁻ appear at 1090, 1014, and 590cm⁻¹. The peak at 1650cm⁻¹ shows the presence of H₂O [54].

G. Adhesion

For evaluating the adhesion of the coated samples we used the scratch tester machine M-TGN80 having diamond intender
landed on the coated substrate producing an indent on the substrate surface up to a maximum load of 60N as shown in Figure 9. The result shows an excellent strength for the coating [56]. The morphological measurements were taken in the stereo microscope under low magnifications (5X) which reveal the distances of the crack from various aspects.

IV. CONCLUSION

Hydroxyapatite is the furtheormost noticeable inorganic substance for biomedical applications. Research showed that natural HAP from bovine bones can be synthesized by using the thermal decomposition method. From this study, no significant difference was observed between bovine bone-derived HAP and naturally occurring HAP. HAP powder electrophoretically deposited on Ti-6Al-4V alloy was used as substrate. The characterization of the coated sample confirmed that the coating enhanced the osseointegration properties of the implant. Moreover, the hardness of the coated samples was observed to be increased up to 35% as compared to the pristine samples. The corrosion rate for coated samples was found to be decreasing from $33.53 \times 10^{-3}$ mpwy to $11.95 \times 10^{-3}$ mpwy. Therefore, the resultant structures show that our produced samples can be used for a wide variety of medical applications with minimal expenses.

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