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Original Research Article

Hydroxyapatite/ Porous silica coating on Cp-Ti substrates for treating dental implant infections

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Abstract

Aim: Modifications to the surface coatings of titanium implants for improving properties like bio-corrosion resistance and anti-bacterial activity are the new area of interest among researchers. This in-vitro study, aimed to evaluate biomechanical and anti-bacterial properties of the titanium surface coated with Hydroxyapatite/porous silica using the wet chemical process.

Materials and methods: In the present study, electrophoretic deposition of a combination of porous silica with hydroxyapatite crystals on the surface of a titanium implant was carried out. The biomechanical properties of the coating were measured using scanning electron micrography(SEM), Fourier-transform infrared (FT-IR), biocorrosion and anti-bacterial activity analysis.

Results: In the present work, electrophoretic deposition of silica with hydroxyapatite-coated titanium has a potential of 30 V. The porous structure is observed by the surface topography analysis. The presence of the functional group verified the FT-IR spectra. The biocorrosion of coated samples was evaluated by SBF solution at the corrosion rate of 0.089 mm/year. The antibacterial activity evaluation of the coated sample prevents the growth of bacteria of S.aureus and E.coli respectively.

Conclusion: This study investigates the electrophoretic deposition of a silica-hydroxyapatite coating on titanium implants for dental infection treatment. The porous coating, confirmed by FT-IR, showed low corrosion and strong antibacterial properties, enhancing cellular adhesion and reducing infection risk, making it suitable for clinical use.

Keywords: Titanium implant, Dental, Silica-HAP, Electrophoretic deposition

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1. Introduction

Modern dental implants are based on osseointegration, a crucial biological process that creates a direct structural and functional bond between the implant material and the surrounding bone. To maximize the osseointegration process, a dental prosthesis is attached after the implant fixture is placed. An abutment is usually placed to promote and facilitate osseointegration prior to this attachment. The quality, thickness, and condition of the surrounding bone and oral tissues have a significant impact on whether an implant succeeds or fails. The mechanical stress that the implant and its structure will experience during routine functional activities, like chewing, is also determined by these factors. Significant impact on the structure of the surrounding bone and its structure will experience during routine functional activities, like chewing, is also determined by these factors.

Because mastication involves large biomechanical forces, the number and location of implants must be carefully considered to ensure the prosthetic system's long-term stability and success.⁷

Metallic biomaterials are frequently employed to satisfy the mechanical strength and stress requirements of dental implants. Particularly, titanium-based metallic implants are perfect for dental applications because of their exceptional mechanical qualities, high specific strength, and biocompatibility.⁷ Even with titanium alloys' advantages, further research and development is still required to create applications that will benefit patients therapeutically. In the manufacturing of medical implants, the use of specific metals

*Corresponding author: Pratibha Ramani Email: suvarnanairk@gmail.com with limited biocompatibility increases the risk of implant failure. Toxic ions and wear debris may be released as a result, and these could build up and enter human tissue, causing health hazards. Numerous surface alterations and cutting-edge technology strategies have been developed to solve these issues, enhance implant performance, and reduce dangers.⁹

Surface modification has been an important strategy in the quest to improve titanium (Ti) implants' functionality. Electrophoretic Deposition (EPD), which provides exact control over the coating thickness and deposition time, is one potential method.¹⁰ Optimizing the implant's interaction with the surrounding bone tissue requires this level of accuracy.

Due to its chemical resemblance to actual bone, hydroxyapatite has been utilized extensively as a bone substitute. For an implant to fuse with native bone effectively, hydroxyapatite coating creates an osteophilic (bone-attracting) surface. However, hydroxyapatite by itself could not yield the best outcomes, which is why silica is added.

A more conducive environment for bone growth and osseointegration is created when silica and hydroxyapatite are combined since it has been demonstrated that this combination increases the activity of osteoblast cells, which are in charge of bone production. Through the development of a highly ordered nanoporous silica layer loaded with hydroxyapatite on a titanium implant, we expanded this idea in our study. This alteration was intended to assess the surface's antibacterial capabilities, which is crucial in reducing surgical site infections, in addition to improving surface characteristics. By employing this novel covering, we want to create implants that are more biocompatible and long-lasting, while also encouraging efficient bone integration and providing defense against bacterial colonization.

2. Materials and Methods

The present study was conducted in the material department of our institution. Since this is a material related study and not implemented in patients/animals, ethical committee approval and informed consents were not required for the conduct of the present study.

2.1. Preparation of Silica/HAp

Tetraethylorthosilicate (TEOS), 0.5 ml, was added to 5 ml of ethanol and continuously stirred for 3 hours at room temperature. Then, 0.3 ml of HCl and 1 ml of H₂O were added in a closed container, and the hydrolysis reaction was carried out at 60°C with continuous stirring for 24 hours. A colloidal silica gel was formed after aging for 12 hours and drying at 500°C in an oven. For the hydroxyapatite solution, 1 M of Ca(NO₃)₂·4H₂O and 0.6 M of H₃PO₄ were dissolved in 50 ml of double-distilled water, with the pH adjusted to 9 using ammonia solution. After adding 0.5 g of porous silica

powder to the hydroxyapatite solution, the mixture was filtered and dried in a hot air oven.

2.2. Preparation of coatings

Commercially pure titanium (Cp-Ti), medical grade, with sample dimensions of $1.5~\rm cm \times 1.5~\rm cm \times 2~mm$ thickness, was purchased from Sigma-Aldrich. The samples were polished with carbon paper up to 1000 grit to remove debris. The polished samples were then cleaned using acetone and distilled water in a high-power ultrasonicator for 10 minutes. Coating was applied using the electrophoretic deposition method at an applied potential of 30 V for 20 minutes. After coating, the samples were dried in a desiccator for characterization analysis.

2.3. Characterization studies

2.3.1. Surface morphology analysis

The coated sample was examined for surface morphological structure utilizing a Field Emission Scanning Electron Microscope equipped with a JEOL Energy Dispersive X-ray Spectrometer (EDS), specifically the JSM-IT800 NANO SEM model.

2.3.2. FT-IR investigations

To identify the functional groups of coated samples, Fourier-Transform Infrared (FT-IR) spectroscopy was performed using the Alpha II Bruker model spectrometer over a wave number range from 4000 to 500 cm-1.

2.4. Electrochemical studies

Biocorrosion studies of implants were performed by the electrochemical studies of corrosion rate. The samples were examined by the Open circuit potential (OCP) and the steady state potential was observed. Potentiodynamic polarization studies were performed by the three-electrode system such as platinum as a counter electrode, reference as a saturated calomel electrode, and working as test material with an exposed area in 1 cm-1. The study was conducted by the electrochemical workstation PGSTAT 302 N Metrohm and it was controlled by a personal computer of NOVA 2.0 software. The polarization resistance was analyzed by Stern Gearmy's equation.¹³

R p =
$$\beta a \times \beta c 2$$
. 3 ×i corr ($\beta a + \beta c$)-----(1)

2.5. Anti-bacterial activity

Two types of bacteria, namely Gram-negative E. coli (ATCC 25922) and Gram-positive S. aureus (ATCC 25923), were used. These bacterial strains were obtained from frozen stock cultures and transferred to Tryptic Soy Agar (TSA). The plates were incubated at 37°C for 18–24 hours. Following incubation, the bacteria were cultured in 50 mL of sterile Tryptic Soy Broth (TSB) with constant agitation at 80 rpm for another 18–24 hours at 37°C. Prior to inoculation, the bacterial strains were subcultured into fresh TSB at a 1:50

ratio and incubated for 2 hours at 37°C with continuous agitation at 80 rpm."

3. Results

3.1. Surface studies

Using SEM-EDX micrographs, the morphology of the film was examined and is displayed in **Figure 1**. The surface morphology reveals the agglomerated structure obtained from the HAP/porous silica-coated Cp-Ti surface, showing a porous nature due to the electrophoretic deposition process, which influences ionic movements. The corresponding EDX profile indicates the presence of O, Ti, Si, Ca, P, and Na across the entire surface. The formation of the coating was evenly distributed throughout the surface. Moreover, the porous morphology facilitated the osseointegration process at the bone-material interface.

3.2. FT-IR Studies

The presence of functional groups was confirmed by the FT-IR spectra shown in **Figure 2**(a). The stretching vibration of the band appeared at 3407 cm⁻¹, indicating the presence of hydroxyl groups (OH). ^{14,15} The peak at 2989 cm⁻¹ was attributed to the methyl group in the sol-gel solution. A stretching vibration of water (OH) molecules was observed at 1612 cm⁻¹. The silica peak at 1023 cm⁻¹ indicates the presence of a Si-O-Si band. ¹⁶ The phosphate (PO₄³⁻) peak was observed at the vibrational mode v₃ of 1023 cm⁻¹, overlapping with the silica particles, along with an additional peak at 787 cm⁻¹. ¹⁷ Overall, the FT-IR spectra confirmed the presence of silica/HAP on the titanium implant.

3.3. Electrochemical studies

3.3.1. Potentiodynamic polarization studies

The potentiodynamic polarization studies of the coated samples were conducted in Simulated Body Fluid (SBF) solution and are displayed in **Figure 2**(b). The Tafel plot was generated with respect to the open circuit potential of ± 250 mV, covering a potential window from -0.5 to 0.29 V. The biocorrosion rate was calculated using the Stern-Genry equation, and the values are provided in **Table 1**. The Tafel plots of both bare and coated samples exhibit corrosion potential (E_corr) values of -0.301 V and -0.201 V, respectively. The coated sample shifts the Tafel plot in the nobler direction by a difference of 100 mV compared to the bare sample, indicating that the coating provides higher resistance. The coated sample demonstrates a low corrosion rate and high polarization resistance. In this regard, the porous silica/HAP coating offers better resistance than the bare sample, making it a suitable option for long-term implant materials in in-vivo applications.

Table 1: Shows the potentiodynamic polarization studies data were evaluated from SBF solutions.

S.No.	Ecorr (V)	I _{corr} (μA cm ⁻²)	Polarization resistance Rp (ohm)	Corrosion rate (mm per year x 10-3)
Bare	-0.301	0.1257	532	1.369
Coating	-0.201	0.0025	2456	0.089

3.4. Anti-bacterial activity

The samples were evaluated using the antimicrobial assay with a zone of inhibition method, and the results are shown in **Figure 3**. The control sample was an antibiotic. The grampositive and gram-negative bacteria exhibited different zones as observed in the results. The coated samples were scratched and dissolved in PBS solution, then added at different concentrations of 50 mg and 100 mg, respectively. **Figure 3** a shows the percentage of the zone of inhibition, indicating greater activity at 100 mg. **Figure 3**b displays the bacterial plates of S. aureus and E. coli, revealing the zones of inhibition. The coated sample resisted strong microbial infections in both gram-positive and gram-negative bacteria, demonstrating its ability to inhibit bacterial growth and its potential use as implants for biomedical applications.

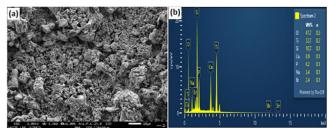


Figure 1: a: SEM image and **b:** EDX profile of HAP/Porous silica coated electrophoretic deposition of Ti implant.

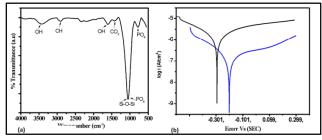


Figure 2: a: FT-IR spectra of porous silica/HAP coated Titanium; b: Tafel plots of bare and coated sample Ti were evaluated in SBF solution.

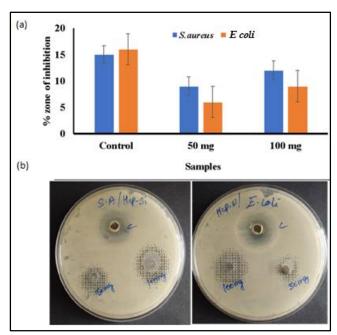


Figure 3: a: Depicts a bar graph with an X-axis showing the different concentrations of S.aureus and E.coli along with the control and Y-axis showing their percentage of inhibition; **b:** Shows the zone of inhibition for S.aureus and E.coli along with control.

4. Discussion

Silica exhibits a variety of advantageous chemical properties, including exceptional thermal resistance, efficient drug release capabilities, and high biocompatibility. These attributes position porous silica-based materials as particularly promising candidates for diverse biomedical applications, such as drug delivery systems and tissue engineering scaffolds. ¹⁸ The unique mesoporous structure of silica plays a crucial role in enabling the controlled release of loaded drugs at targeted sites within the body, enhancing therapeutic efficacy while reducing potential side effects.

The advancement of composite coatings, especially those that combine silica and zinc oxide, has shown significant performance benefits. During the electrophoretic deposition process, conducted at voltages ranging from 70 to 80 V for various durations, these materials develop a dense morphology. This dense coating enhances corrosion resistance, an essential property for implants, owing to its strong adhesion to the substrate, which is vital for maintaining implant integrity in biological environments.¹⁹

Additionally, Rau et al.²⁰ investigated the pulsed laser deposition method to create a silicon-substituted hydroxyapatite (Si-HA) coating on titanium implants, using a composition of 1.4 wt% silicon. This innovative technique produces dense layers that exhibit improved mechanical strength and a high degree of crystallinity. Such structural improvements are critical, as they promote better osteoblast adhesion—the cells responsible for bone formation—and facilitate increased bioapatite precipitation. This precipitation

is especially advantageous because it increases the roughness of the coating, which improves the conditions for cell adhesion and development. Our findings corroborate this, showing that the coated implants' porous surface shape promotes osteoblast adhesion and thus improves integration with bone tissue.

The non-toxicity, non-immunogenic, and biocompatible qualities of hydroxyapatite (HAP) make it an especially desirable substance. Its innate antibacterial properties make it more appropriate for use in biomedical settings, particularly when preventing infections is crucial. Our research showed that the sponge-ceramic coating had strong antibacterial activity at 50 μ g/mL, with inhibition zones of 13 mm against E. coli and 16 mm against S. aureus. This efficacy is essential because it not only helps shield implants from infections but also fosters a more wholesome healing environment.

Building on these positive results, we anticipate that the electrophoretic deposition of coatings containing calcium silicates and Sr-Zn-Mg-Ce minerals produced from sponges will significantly improve bone regeneration processes. ²¹ This technique involves first loading antibiotics like ciprofloxacin or gentamicin sulfate onto pristine hydroxyapatite nanoparticles. After that, they go through a single-step electrophoretic deposition process to create osteoconductive and antimicrobial nanoparticles with long-lasting drug release. These coatings are positioned as revolutionary solutions in clinical settings because of their dual functionality, which promotes bone regeneration while offering antibacterial protection.

Applying hydroxyapatite/silica composites in clinical settings has the potential to transform the treatment of implant-related infections and greatly increase their effectiveness across a range of biomedical applications, as research in this field continues to advance. This research attempts to establish new standards for the functionality and design of biomedical implants in addition to improving patient outcomes.

5. Conclusion

The present study investigates the electrophoretic deposition of a silica-hydroxyapatite composite coating on titanium implants intended for the treatment of dental infections. The electrophoretic deposition process resulted in a surface exhibiting a distinct porous morphology, which was further characterized by Fourier-transform infrared (FT-IR) spectroscopy to confirm the presence of functional groups. Corrosion studies revealed that the coated samples exhibited a corrosion rate of 0.089 mm/year, indicating their suitability for long-term implant applications. Additionally, the antibacterial activity of the coated samples demonstrated significant inhibition of bacterial growth at the implant site, thereby reducing the potential for infection. The porous nature of the coating enhances cellular adhesion following

implantation, making it advantageous for both dental and broader clinical applications.

6. Sources of Funding

None.

7. Conflict of Interest

None.

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