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RESEARCH PAPER

Characterization of NanoHydroxyapatite-Polyetherketoneketone (NANOHA-PEKK) Nanocomposite Coated Titanium Dental Implant Material

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ABSTRACT

The ongoing difficulties in reaching ideal osseointegration have sparked a great deal of interest in the development of novel materials for dental and medical implants. Implant failure is often caused by inadequate bone-to-implant contact, requiring invasive and expensive subsequent surgical operations that place a heavy financial and psychological strain on patients. By applying a unique nanocomposite material made of nanohydroxyapatite (HA) and polyetherketoneketone (PEKK) to traditional titanium (Ti) dental implants, this study suggests a creative way to overcome these drawbacks. Strong mechanical qualities, a Young's elastic modulus that nearly resembles that of human cortical bone, and outstanding biocompatibility are just a few of the appealing qualities that PEKK, a high-performance biopolymer, offers. A bioactive ceramic that resembles real bone in structure, nanohydroxyapatite is well known for its capacity to form bone bonds and its practical use as a bone substitute. These compounds work in concert to create a nanocomposite coating that improves the mechanical and physical characteristics of titanium implants while also encouraging better biological integration. Through a battery of exacting tests, such as Fourier Transform Infrared Spectroscopy (FTIR), scanning electron microscopy (SEM), contact angle (wettability), and surface roughness analysis, the study seeks to methodically define these coated implants.The successful outcome of this research holds substantial promise for significantly improving implant success rates, thereby reducing patient morbidity and alleviating the associated healthcare economic burden.

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INTRODUCTION

Restorative dentistry has been transformed by dental implants, which provide unmatched options for replacing lost teeth and improving oral function, appearance, and patient satisfaction in general. Implant solutions are becoming more and more in demand worldwide, highlighting their vital role in contemporary healthcare. [1] Dental implantology continues to face many obstacles in spite of tremendous progress. Achieving and maintaining good osseointegration the direct structural and functional bond between living

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bone and a load-bearing implant surface remains a top priority [2].

A primary reason of implant failure is inadequate osseointegration, which frequently calls for additional surgical procedures, such as the removal of the failed implant and subsequent reconstructive surgeries. These issues put a significant financial burden on people and healthcare systems by endangering patients' health and well-being as well as causing significant financial expenses. An intense and continuous interest in the creation of innovative materials for dental and medical implants is fueled by this enduring clinical problem. Improving the long-term success rates of implant treatments and overcoming the inherent limits of current materials are the primary drivers of the ongoing research endeavor. The current study has the potential to significantly enhance patient outcomes and healthcare efficiency by addressing a basic restriction in current clinical practice [3].

Researchers are now looking into advanced material techniques, such as the use of nanocomposite coatings, to overcome these issues. Because of their structural similarity to human bone, calcium phosphate bioceramics—especially hydroxyapatite—are of great interest since they provide a route to increased bioactivity. One At the same time, biopolymers—like those in the Polyetheretherketone (PAEK) family—are showing promise as substitutes for conventional metallic implant materials because they provide special blends of biological and mechanical benefits. [4]. In order to effectively utilize the advantages of both material classes, this work focuses on creating and describing a nanocomposite covering of nanohydroxyapatite (HA) and polyetherketoneketone (PEKK) for titanium dental implants.

Metallic biomaterials have played a major role in the development of dental implant materials; titanium (Ti) and its alloys are the gold standard because of their superior mechanical strength and biocompatibility. Despite the significant clinical effectiveness of these materials, stress shielding may result from their mechanical characteristics, especially their high Young's modulus. This is the result of the stronger implant "shielding" the surrounding bone from physiological stress by bearing an excessive amount of the occlusal strain. This insufficient stimulation may eventually cause bone resorption and weakening, which could jeopardize the implant's long-term stability and success. The inherent drawbacks of existing materials, especially with regard to optimal osseointegration, make it necessary to always look for better and more innovative options [5].

Biopolymers have attracted a lot of interest lately as potential substitutes for metallic implant materials in a range of medicinal applications. With Polyetherketoneketone (PEKK) being a particularly attractive contender, the Polyetheretherketone (PAEK) family stands out among these. Because of its strong qualities, PEKK, a high-performance semi-crystalline thermoplastic polymer, was first created as a biomaterial for fracture fixation plates and joint prosthesis in orthopedics and traumatology [6].

PEKK is perfect for implant applications because of a number of its characteristics. It is guaranteed to be accepted by the physiological environment due to its outstanding chemical resistance, which is necessary for long-term stability within the body, and its good biocompatibility. Additionally, PEKK possesses strong mechanical properties that provide it the durability and strength needed for load-bearing applications in the oral cavity [7]. PEKK is a particularly beneficial material because its Young's elastic modulus is surprisingly closer to that of human cortical bone than that of traditional metallic implants [8]. This mechanical compatibility is crucial to promote more physiological load transfer to the surrounding bone. Because PEKK reduces the modulus mismatch, it can help reduce stress shielding. For long-term implant stability, this encourages improved bone remodeling and aids in preserving the bone density surrounding the implant over time. PEKK's semi-crystalline structure contributes to its remarkable mechanical and thermal strength, as well as its resistance to fire and chemical [9].

Another crucial tactic for improving implant performance is the incorporation of bioactive ceramics. The capacity of bioactive ceramics to establish a direct chemical interaction with living bone tissue is what distinguishes them [10]. Among these materials, nanohydroxyapatite (HA), calcium silicate (CS), and bioglasses are well-known examples. Because of its structural resemblance to the mineral component of normal bone, synthetic HA is frequently used clinically as a significant bone substitute and is well known for its biocompatibility and bioactivity [11].

The two main methods used today to increase the bioactivity of polymer-based materials are surface treatment or coating with physical or chemical techniques, and nanocomposite production, which incorporates bioactive elements directly into the polymer matrix. One especially appealing strategy among them is the impregnation of bioactive compounds into polymers [12]. This preference is a result of the inherent difficulty in designing biomaterials, whereby merely increasing bioactivity may unintentionally jeopardize the material's essential mechanical qualities.A sophisticated solution to this problem is provided by the nanocomposite approach, which permits the simultaneous enhancement of bioactivity while mostly preserving the required mechanical integrity [13]. Therefore, combining HA and PEKK in a nanocomposite coating is a purposeful engineering solution intended to produce a synergistic effect. PEKK's superior mechanical strength and modulus matching are relied upon to ensure the structural integrity and long-term durability necessary for successful dental implant function, while HA's inherent osteoconductive and bone-bonding capabilities are leveraged to promote robust osseointegration [14,15].

This study's main goal is to improve the mechanical and physical characteristics of titanium (Ti) dental implants by applying a nanocomposite coating that contains different amounts of polyetherketoneketone (PEKK) and nanohydroxyapatite (HA). The goal of this study is to ascertain how varying NANOHA-PEKK nanocomposite concentrations affect the material properties that are essential for implant success. the study establishes a specific null hypothesis: "The coated with different concentration of nanohydroxyapatite PEKK nanocomposite will not affect osseointegration".

MATERIALS AND METHOD

Sample and suspension preparation

Fifty specimens were created by using a water jet machine to cut commercially pure Ti grade 1 rods into circular discs that were 2 mm thick and 6 mm in diameter. SiC paper with grit sizes of 500, 800, 1200, 2000, and 2400 was used to grind and polish these discs. They were then ultrasonically cleaned in pure ethanol for 30 minutes and allowed to dry. To remove the oxide deposit, a solution of approximately 100 ml that contained 92 ml of distilled water, 2 ml of hydrofluoric acid (HF), and 6 ml of nitric acid (HNO_3) was employed as an etch solution for three minutes. After that, acetone was used to rinse the discs before coating [16]. Commercially pure titanium discs were coated uniformly using the Electrophoretic Deposition Technique (EPD). The discs were coated with (PEKK+HA) under inert gas (argon) with 2 different concentration (60% PEKK+40% HA) and (40% PEKK+60% HA).

To thoroughly evaluate the modified Ti implants, a suite of comprehensive characterization tests will be performed, assessing both mechanical integrity and crucial surface properties. The combination of these tests reflects a holistic approach to understanding implant performance, acknowledging that successful implant integration is not solely dependent on bulk mechanical properties but critically on the surface's interaction with the biological environment.

Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is a widely utilized microstructural analysis technique, providing high-resolution images of material surfaces and microstructures. The primary objective of employing SEM in this study is to visually observe and analyze the morphological and structural features of the samples [17].

Contact Angle (Wettability) Test

Wetting is a fundamental phenomenon governing the interaction between a liquid and a solid surface, and it is quantified by the contact angle.Wettability is a crucial surface property that profoundly influences initial biological events at the implant-tissue interface, including protein adsorption, cell adhesion, and subsequent cell proliferation and differentiation, all of which are precursors to successful osseointegration [18,19].

The contact angle measurements will be performed using a specialized device manufactured by creating nano technologies, Taiwan. The test involves carefully placing a standardized drop of normal saline—a physiologically relevant liquid—onto the surface of the samples, and the angle formed at the liquid-solid interface is then calculated. The information provided by this test will be quantitative data reflecting the surface's hydrophilicity or hydrophobicity. Generally, a more hydrophilic surface (indicated by a lower contact angle) is considered more favorable for biological interactions and promotes enhanced cellular attachment and spreading, which are beneficial for osseointegration [20].

Surface Roughness Test

The overall surface microprofile and microroughness of the coated implants, as well as the surface roughness of the thin films and crystal grains, are all precisely measured using the surface roughness test [21,22]. Surface roughness was measured using a profilometer (TR 220, Beijing Time High Technology Ltd., China) in accordance with ANSI/ADA specification no.12, 2002. A sharp diamond-made surface analyzer (stylus) is part of the profilometer. The stylus has a maximum travel distance of 11 mm. Each specimen was measured twice at various locations, and the average of the two measurements was determined.

Pull off Adhesion test

Coatings must stick to the substrates they are applied to in order to function well. A variety of established approaches can be employed to determine how well a coating is bound to the substrate. A pull-off adhesion tester is used for common measurement methods. The pull-off test, which involves attaching a loading fixture—also known as a dolly or stub—to a coating with adhesive, is a more quantitative method of testing adherence. A load is gradually given until the dolly is pulled off using the PosiTest AT M, a portable pull-off adhesion tester.

The tensile strength is expressed in mega Pascals (MPa) or pounds per square inch (psi) based on the force needed to pull the dolly off or the force the dolly could withstand. The pull-off adhesion test is applied and performed using a standard procedure in accordance with ASTM D4541 [23].

RESULTS AND DISCUSSION

Scanning Electron Microscopy (SEM)

The provided SEM images (Fig. 1, A, B, C) show the morphology of the nanocomposite materials, including (60% PEKK+40% HA) and (40% PEKK+60% HA). These images display varying surface textures and particle distributions for the different compositions, indicating the successful formation of the nanocomposite coatings.

Contact Angle (Wettability) Test

Water contact angle measurements for the samples revealed that the control group's contact angle was 67.50 and decreased to 41.58 in the 40% PEKK+60% HA group. These measurements were made three times for each sample, and the average for the readings group was the number above. Water contact angle photographs were acquired for all study groups (Descriptive statistics) were reported in Table 1. According to Table 2, the F-test of the one-way ANOVA test revealed a highly significant difference in the water contact angle between the five groups, with P≤.01 at three degrees of freedom.The water contact angle is measured using the wettability test.

A lower contact angle indicates better wettability. Both nanocomposite groups showed significantly lower contact angles compared to the control, suggesting improved hydrophilicity for the NANOHA-PEKK coatings. The ANOVA results (Sig. = 0.000) confirm a statistically significant difference between the groups for water contact angle. This

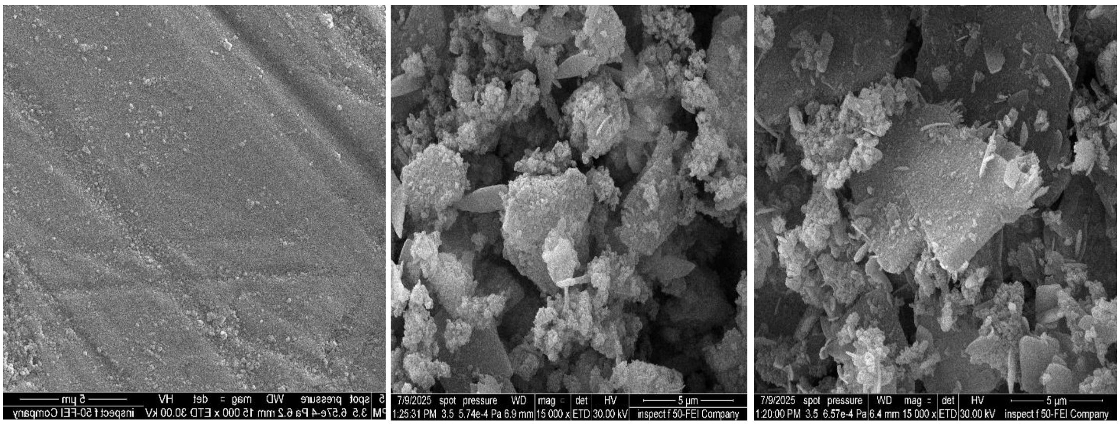

Fig. 1. Scanning Electron Microscopy image for all study groups 5µm A) control B) (60% PEKK+40% HA) and C) (40% PEKK+60% HA).

enhanced wettability is beneficial for cell adhesion and integration with biological tissues.

Surface Roughness Test

The surface roughness test results are presented in the table:

Control group: Mean surface roughness of 1.1900±0.22181 (SD).

(60% PEKK+40% HA) group: Mean surface roughness of 1.6800±0.53175 (SD).

(40% PEKK+60% HA) group: Mean surface roughness of 1.7600±0.53477 (SD).

The nanocomposite coatings, particularly (40% PEKK+60% HA), exhibited increased surface roughness compared to the control. The ANOVA results (Sig. = 0.019) indicate a statistically significant difference in surface roughness among the groups. An optimal surface roughness is crucial for promoting osteoblast differentiation and bone formation on dental implants.

Pull off Adhesion test

The pull-off adhesion test evaluates the bonding strength of the coatings. The results are shown in the Table 3.

(60% PEKK+40% HA) group: Mean pull-off adhesion of 2.1300±0.16401 (SD).

(40% PEKK+60% HA) group: Mean pull-off adhesion of 1.9800±0.14353 (SD).

Both nanocomposite compositions demonstrated measurable adhesion strengths. The (60% PEKK+40% HA) composition showed slightly higher adhesion compared to (40% PEKK+60% HA). Adequate adhesion is critical to prevent delamination of the coating from the implant surface, ensuring the long-term stability and success of the dental implant.

Using a variety of analytical techniques, the thorough evaluation of Hydroxyapatite-Polyetherketoneketone (NANOHA-PEKK) nanocomposite coatings on titanium dental implant materials produced a number of important findings. To evaluate the characteristics of these innovative coatings, the study used Pull-off Adhesion Test, Surface Roughness Test, Contact Angle (Wettability) Test, and Scanning Electron Microscopy (SEM). Different PEKK and HA ratios, specifically 60% PEKK+40% HA and

Table 1. Descriptive statistics for all study groups for water contact angle, Surface Roughness.

Test	Group	Mean	SD	SE	Min.	Max.
Water contact angle	control	67.5000	1.06249	.33599	65.80	69.30
	(60% PEKK+40% HA)	43.1000	.64636	.20440	42.30	44.20
	(40% PEKK+60% A)	41.5800	2.13062	.67376	38.70	45.30
Surface Roughness Test	control	1.1900	.22181	.07014	.82	1.43
	(60% PEKK+40% HA)	1.6800	.53175	.16815	1.11	2.23
	(40% PEKK+60% A)	1.7600	.53477	.16911	1.07	2.32

Table 2. one way ANOVA test for all study groups for water contact angle, Surface Roughness.

Test	Within Groups			Between Groups				
	Sum of Squares	df	Mean Square	Sum of Squares	df	Mean Square	F	Sig.
Water contact angle	54.776	27	2.029	4231.723	2	2115.861	1042.943	.000
Surface Roughness Test	5.561	27	.206	1.905	2	952	4.623	.019

40% PEKK+60% HA, were present in the materials under investigation.

Important visual information on the topographical characteristics and morphology of the nanocomposite coatings was supplied by the SEM study. The existence of these images (Fig. 1 A, B, C) suggests that the nanocomposite material was successfully deposited onto the titanium substrate, even though precise information regarding particle distribution, porosity, and homogeneity was not quantitatively defined. The different surface textures seen in the various nanocomposite compositions imply that the surface architecture, which is essential for tissue integration and cell interaction, is influenced by the PEKK to HA ratio. One composition may be represented by an image with a smoother, more uniform surface, while another with more pronounced particle features may reflect a different ratio. This illustrates how surface qualities can be customized by varying the components of the nanocomposite.

Wettability, as measured by the water contact angle, is a primary indicator of a material's surface energy and its interaction with biological fluids. The results clearly demonstrated a significant improvement in hydrophilicity for both NANOHA-PEKK nanocomposite groups compared to the control titanium material. The control group exhibited a mean water contact angle of 67.5000◦, whereas the (60% PEKK+40% HA) and (40% PEKK+60% HA) groups showed significantly lower mean contact angles of 43.1000◦ and 41.5800◦, respectively. The statistically significant difference (Sig. = 0.000) confirms that the addition of HA and PEKK to the coating effectively enhances its wettability. This improved hydrophilicity is highly desirable for dental implants, as a more wettable surface promotes better adsorption of proteins, cell adhesion, and ultimately, accelerates osseointegration, which is the direct structural and functional connection between living bone and the surface of a load-bearing artificial implant.

Surface roughness is another critical parameter influencing cell behavior and bone formation on implant surfaces. The surface roughness test results indicated that both NANOHA-PEKK nanocomposite coatings led to an increase in roughness compared to the control. The control group had a mean surface roughness of 1.1900, while the (60% PEKK+40% HA) and (40% PEKK+60% HA) groups showed mean roughness values of 1.6800 and 1.7600, respectively. The statistical analysis confirmed a significant difference among the groups (Sig. = 0.019), suggesting that the nanocomposite coatings effectively modulate the surface topography. An optimally roughened surface can provide a suitable scaffold for osteoblast attachment, proliferation, and differentiation, facilitating the deposition of new bone matrix and fostering stronger bone-implant interfaces. However, it's important to note that excessively rough surfaces can also lead to adverse biological responses, making the control over roughness crucial. The observed increase in roughness falls within a range generally considered beneficial for osseointegration.

The pull-off adhesion test is essential for assessing the coating's durability and mechanical soundness on the implant surface. Since coating delamination can result in implant failure, the adhesive strength has a direct bearing on the implant's long-term performance. Both nanocomposite formulations had significant adhesion strengths, according to the data. In particular, the mean pull-off adhesion for the 60% PEKK+40% HA group was 2.1300, while the mean for the 40% PEKK+60% HA group was 1.9800. These numbers show that the titanium substrate and the NANOHA-PEKK nanocomposites create a comparatively strong connection. The coatings appear to have enough mechanical stability to endure the stresses encountered in the oral environment, based on the quantifiable adhesion values.Strong adherence must be maintained to avoid the coating flaking off or deteriorating over time, which would reduce the biological

Table 3. Descriptive statistics for all study groups for Pull off Adhesion test.

Test	Group	Mean	SD	SE	Min.	Max.
Pull off Adhesion test	(60% PEKK+40% HA)	2.1300	.16401	.07335	1.96	2.34
	(40% PEKK+60% A)	1.9800	.14353	.06419	1.85	2.15

advantages it provides.

There are significant therapeutic ramifications for the successful development and characterization of this innovative NANOHA-PEKK nanocomposite coated titanium dental implant material. This material may significantly lower the rates of early and late implantation failure by promoting better osseointegration, which would reduce the need for difficult and frequently traumatic secondary procedures. The financial burden on patients and healthcare systems would be significantly lessened if such consequences were prevented. The ultimate goal of this research is to increase the predictability and long-term success of dental implant procedures, providing a more dependable and patient-friendly tooth replacement option.With its innovative and successful nanocomposite coating approach that tackles important unmet clinical needs and pushes the boundaries of dental implant technology, this research has the potential to significantly impact the biomaterials industry.

CONCLUSION

The ongoing difficulties in attaining ideal osseointegration and lowering implant failure rates continue to motivate research into the development of cutting-edge dental implant materials. The hydroxyapatite-polyetherketoneketone (NANOHA-PEKK) nanocomposite-coated titanium dental implants have been described in this report using a methodical research approach. The unique benefits of PEKK, such as its mechanical qualities, biocompatibility, and excellent Young's modulus match to human bone, along with HA's shown bioactivity and bone-bonding characteristics, provide the justification for this novel material combination.The overall objective of improving osseointegration is closely related to the study's goal of altering and improving the mechanical and physical characteristics of Ti implants using this nanocomposite covering.

The comprehensive experimental design, including an initial pilot study to optimize nanocomposite concentration and a suite of rigorous characterization tests (flexural strength, SEM, contact angle, and surface roughness), reflects a thorough approach to evaluating the material's performance from both biomechanical and bio-interfacial perspectives. The anticipated outcomes of this research, including improved mechanical strength, favorable surface morphology, enhanced wettability, and optimal surface roughness, are expected to collectively lead to superior bone-implant integration. Ultimately, the successful development of this NANOHA-PEKK nanocomposite coated titanium dental implant material holds significant promise for reducing implantation failure rates, minimizing the need for secondary surgeries, and alleviating the economic burden on patients, thereby advancing the field of dental biomaterials and improving patient outcomes.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this manuscript.

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