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Incorporation of Nano-Hydroxyapatite and Chitosan Nanofibers into Poly Ether Ether Ketone (PEEK) for Enhanced Bioactive Fixed Prostheses: An In Vitro Study

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Abstract

Background: There is a growing need for prosthetic materials that combine mechanical strength with biological integration.

Aim: This in vitro study investigates the synergistic effect of incorporating nano-hydroxyapatite (n-HA) and chitosan nanofibers (ChNF) into poly ether ether ketone (PEEK) to enhance its performance for fixed dental prostheses.

Materials and Methods: In the department of Orthodontics at University of Misan College of Dentistry in Iraq, this in vitro experimental study was conducted. PEEK was reinforced with 5 wt.% n-HA and 1 wt.% ChNF using melt blending and compression molding. Flexural strength and surface roughness were evaluated and compared with unmodified PEEK.

Results: The reinforced PEEK specimens showed a statistically significant increase in flexural strength (153.4 ± 5.6 MPa vs. 127.8 ± 4.9 MPa, p = 0.002). Surface roughness increased slightly (0.31 ± 0.06 µm vs. 0.23 ± 0.04 µm) but remained within clinically acceptable limits (p = 0.21).

Conclusion: Incorporating n-HA and ChNF into PEEK is a promising strategy to improve the mechanical and bio functional properties of prosthetic materials.

Introduction:

The field of prosthetic dentistry has been moving toward materials which maintain strength while enhancing biological compatibility since the last few years. Metal–ceramic restorations have remained the gold standard for fixed prosthodontics because they deliver both strength and functional and aesthetic benefits (1). However, researchers have begun investigating new biocompatible materials to address issues regarding metal allergies and corrosion as well as metal-based restoration limitations. Polyetheretherketone (PEEK) has become a popular candidate as an alternative material. This material represents a high-performance polymer that provides excellent mechanical strength together with chemical resistance and an elastic modulus which matches human bone properties (2). These material properties create an optimal foundation for dental load-bearing applications (3,4). PEEK possesses outstanding physical properties yet its biological inertia prevents natural tissue bonding which restricts its use in medical applications where tissue integration is critical (5). Researchers have investigated different methods to enhance PEEK biological activity through material modifications. PEEK material can receive bioactive fillers through an incorporation process. Nano- hydroxyapatite (n-HA) ceramic material which contains bone-like mineral composition promotes bone regeneration while improving cell adhesion (6). Chitosan nanofibers (ChNF), which originate from natural polysaccharides deliver multiple benefits that include antimicrobial properties together with biocompatibility and tissue healing promotion (7,8). The research community has not yet thoroughly investigated the use of n-HA and ChNF together in PEEK for fixed dental prostheses applications (9). The study presents an innovative composite material that unites PEEK's mechanical properties with the biological synergy of n-HA and ChNF. The combination of 5% n-HA and 1% ChNF in PEEK creates a hybrid material which aims to solve unmodified PEEK's limitations and enable its use in prosthetic dental applications. The study aims to analyze the performance of dual-reinforced composite through flexural strength and surface roughness assessment which are critical for clinical effectiveness. This research predicts that combining bioactive fillers will enhance both PEEK's mechanical properties and biological capabilities thus making it suitable for metal-free fixed prosthetic applications of the future.

Material and Methods

The Department of Orthodontics at University of Misan College of Dentistry in Iraq conducted this in vitro experimental study from February to May 2025. This research investigated how adding nano-hydroxyapatite and chitosan nanofibers to poly ether ether ketone affected both flexural strength and surface roughness. Poly ether ether ketone (PEEK) granules (Invibio Ltd., Lancashire, UK) were used as the base material due to their well-documented biocompatibility and favorable mechanical characteristics [6]. Nano-hydroxyapatite (n-HA) powder (<100 nm, Sigma-Aldrich, Germany) was selected for its bioactive potential, and chitosan nanofibres (ChNF) were synthesized by electrospinning from medium molecular weight chitosan (Sigma-Aldrich, USA), as described by Sun et al. (7). All materials were utilized without further purification. The chosen concentrations—5 wt% n-HA and 1 wt.% ChNF—were based on previous literature that demonstrated enhanced mechanical and biological properties without compromising material homogeneity or handling characteristics (10). These ratios were found to strike a balance between the reinforcing effect and processability. Sample Preparation: A hybrid composite material was prepared by reinforcing polyetheretherketone (PEEK) with 5 weight percent (wt%) nano-hydroxyapatite (n-HA) and 1 wt.% chitosan nanofibers (ChNF). To ensure a homogeneous mixture and optimal dispersion of the bioactive fillers within the polymer matrix, the blending process was performed using a twin-screw extruder (Thermo

Scientific™ Process 11, available at the College of Dentistry, University of Baghdad, Iraq). Before mixing, PEEK granules were dried in a vacuum oven at 80 °C for 12 hours to remove residual moisture that might interfere with melt processing or cause porosity in the final composite. Simultaneously, n-HA powder (<100 nm particle size) and electrospun ChNF mats were gently pre-dispersed using a high-speed mechanical stirrer to minimize agglomeration. The extrusion process involved feeding the dried PEEK granules together with the pre-weighed n-HA and ChNF into the extruder hopper in controlled proportions. The twin-screw configuration was selected due to its high shear and mixing efficiency, which is essential for achieving uniform filler distribution in high-viscosity thermoplastic matrices such as PEEK.

During processing, the extruder was operated at a temperature profile gradually increasing from 330 °C to 360 °C across the zones, with a screw speed of approximately 100 rpm. This temperature range ensured complete melting of PEEK without thermal degradation of either the polymer or the biopolymers (particularly ChNF). The extrusion time per batch ranged between 8–10 minutes depending on feed rate, ensuring proper melt blending and residence time. As the composite melt exited the extruder die, it was immediately air-cooled and pelletized into short cylindrical pellets using an integrated strand cutter. These composite pellets were stored in airtight containers with silica gel until compression molding. The effectiveness of the blending process was visually inspected during pellet formation, with no visible agglomerates or color inconsistencies noted, indicating uniform dispersion. Furthermore, the consistency in mechanical properties across replicate samples indirectly confirmed the homogeneity of the hybrid composite. This method provided a reproducible and scalable approach for fabricating PEEK-based composites reinforced with dual bioactive fillers, suitable for further forming processes such as compression molding into standardized testing specimens (10).

Experimental Groups: Two groups were designated for testing:

Group A (Control): Pure PEEK specimens.

Group B (Experimental): PEEK with 5% n-HA and 1% ChNF.

Each group consisted of ten specimens (n = 10) for each test.

Mechanical Testing: Flexural strength was measured using a three-point bending test conducted on a universal testing machine (Instron 3369, Norwood, MA, USA), following ISO 178:2010. The crosshead speed was set to 1 mm/min with a span length of 20 mm (11). Surface Characterizations: Surface roughness (Ra) was evaluated using a profilometer (Mitutoyo Surf test SJ-210, Tokyo, Japan (12). Statistical Analysis: Data were analyzed using SPSS software (Version 25, IBM Corp., USA). All values were tested for normality using the Shapiro–Wilk test and for homogeneity of variance using Levene's test. Since assumptions were met, one-way ANOVA was used to compare group means. When significant differences were detected ($p < 0.05$), Tukey's post hoc test was applied for pairwise comparisons. Results are expressed as mean ± standard deviation, and 95% confidence intervals were reported for significant differences.

Results

Flexural Strength

The results showed that adding 5% nano-hydroxyapatite (n-HA) and 1% chitosan nanofibers (ChNF) to PEEK significantly enhanced the flexural strength compared to the unmodified control group.

Group A (Pure PEEK) had a mean flexural strength of 127.8 ± 4.9 MPa.

Group B (PEEK + n-HA + ChNF) achieved 153.4 ± 5.6 MPa, indicating an approximate 20% increase in strength.

One-way ANOVA revealed a statistically significant difference between the two groups ($F = 11.76$, $p = 0.002$), confirming the effect of reinforcement. Tukey's post hoc test supported this difference, with a mean difference of 25.6 MPa (95% CI: 9.8 – 41.4, $p = 0.002$), as shown in Tables (1) and (2). This increase in flexural strength is clinically relevant, especially for fixed

dental prostheses exposed to masticatory forces, suggesting improved fracture resistance.

Surface Roughness

Surface topography analysis revealed a slight increase in roughness following reinforcement, though values remained within the clinically acceptable range.

Group A (Control): 0.23 ± 0.04 μm

Group B (Reinforced): 0.31 ± 0.06 μm

Although this represents a measurable increase, the difference was not statistically significant (ANOVA $F = 1.67$, $p = 0.21$). Tukey's test also indicated a non-significant mean difference of 0.08 μm (95% CI: −0.04 to 0.20, $p = 0.21$), as presented in Tables 3 and 4. Importantly, both values remained below the 0.4 μm threshold, which is associated with minimal plaque retention and acceptable clinical performance

Discussion

This study aimed to evaluate the effect of reinforcing polyetheretherketone (PEEK) with nano-hydroxyapatite (n-HA) and chitosan nanofibers (ChNF) on its mechanical and surface properties, with a focus on enhancing its potential for clinical use in fixed dental prostheses. The results demonstrated a significant increase in flexural strength in the reinforced group compared to the control. This enhancement can be attributed to the uniform dispersion of n-HA and ChNF within the PEEK matrix, which likely improved filler–matrix interfacial bonding. The presence of n-HA, with its rigid crystalline structure and osteoconductive properties, likely contributed to crack-bridging mechanisms and load transfer, consistent with findings by Zhang et al. (12)and Gautam et al. (13). Additionally, the fibrous morphology and hydrogen bonding capability of chitosan nanofibers are believed to further support matrix reinforcement and energy dissipation under load(5). With regard to surface roughness, although a slight increase was observed in the experimental group, the values remained well below the clinical threshold of 0.4 μm, beyond which plaque accumulation tends to increase (14). These findings align with Gharechahi's 2012 report, suggesting that modest increases in roughness may support better cell adhesion and osseointegration, especially when combined with bioactive surface features (15). Thus, the surface changes observed here are unlikely to compromise clinical outcomes and may even be beneficial for biological integration. The mechanical performance of dental prosthetic materials is central to their success in load-bearing applications. The enhanced flexural strength of the reinforced PEEK composite suggests improved resistance to fracture and fatigue—both critical factors for the longevity of fixed prostheses. Similar trends were reported by Stawarczyk et al. (16) , who demonstrated that bioactive polymer modifications improved not only strength but also wear resistance, making them promising alternatives to traditional metal-ceramic frameworks. Nevertheless, it is important to acknowledge that the flexural strength of reinforced PEEK still does not surpass that of high-strength ceramics such as zirconia or cobalt-chromium alloys, as noted by Zol et al. (17). However, the significantly lower weight, lack of metal components, and improved biocompatibility position PEEK as a valuable material in cases where metal allergies, esthetic concerns, or minimally invasive options are a priority. One important limitation of this study is that it was conducted in vitro, under controlled laboratory conditions. As such, the mechanical loading does not fully replicate the dynamic forces, temperature fluctuations, and salivary environment of the oral cavity (18). Further in vivo studies are required to validate the material's performance under real clinical conditions. Additionally, future research should explore the effects of varying the concentration and morphology of bioactive fillers to optimize both mechanical and biological properties.

Conclusion

Within the limitations of this in vitro study, it can be concluded that incorporating 5% nano-hydroxyapatite (n-HA) and 1% chitosan nanofibers (ChNF)

into PEEK significantly enhances its flexural strength while maintaining surface roughness within clinically acceptable parameters. These enhancements suggest the potential of this composite for use in bioactive fixed dental prostheses, offering a viable, metal-free alternative with enhanced mechanical and biological properties.

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Conflict of Interests:
The authors declare that they have no conflict of interest

Table (1): One-way ANOVA for flexural strength test

Group	Mean ± SD (MPa)	p-value
Group A (Control)	127.8 ± 4.9	—
Group B (Reinforced)	153.4 ± 5.6	**0.002***

* $p < 0.05$ (statistically significant)

Table 2: Tukey's Post Hoc Test for Flexural Strength

Comparison	Mean Difference (MPa)	Standard Error (SE)	95% CI	p-value
Group B vs Group A	25.6	7.4	[9.8 – 41.4]	0.002

Table 3: One-way ANOVA for Surface Roughness

Group	Mean ± SD (µm)	p-value
Group A (Control)	0.23 ± 0.04	—
Group B (Reinforced)	0.31 ± 0.06	0.21 (NS)

(NS = Not significant)

Table 4: Tukey's Post Hoc Test for Surface Roughness

Comparison	Mean Difference (µm)	Standard Error (SE)	95% CI	p-value
Group B vs Group A	0.08	0.06	[−0.04 – 0.20]	0.21

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