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RESEARCH ARTICLE
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DEVELOPMENT AND EVALUATION OF NANO-HYDROXYAPATITE AND SILICA-REINFORCED DENTAL COMPOSITES: ENHANCING MECHANICAL STRENGTH AND HYDROLYTIC STABILITY

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Abstract

Background: Dental composites are widely used restorative materials; however, their mechanical properties, wear resistance, and hydrolytic stability remain key challenges. Nano-hydroxyapatite (HAp) and silica nanoparticles have shown potential to enhance composite performance by improving strength, durability, and resistance to degradation.

Objective: This study aimed to develop and evaluate novel dental composites incorporating nano-HAp and silica fillers to enhance mechanical strength, wear resistance, and hydrolytic stability.

Methodology: Dental composite samples were fabricated with 0%, 5%, 10%, and 15% nano-HAp-silica filler content. Flexural strength, wear resistance, water sorption, and solubility were assessed following ISO 4049 standards. Mechanical testing was performed at UET Peshawar, and hydrolytic stability was analyzed at Women Dental College, Abbottabad. Statistical analysis was conducted using ANOVA with post-hoc Tukey's test ($p < 0.05$).

Results: The incorporation of nano-fillers significantly improved mechanical properties ($p < 0.001$). Flexural strength increased from 78.5 MPa (control) to 110.8 MPa (15% filler composite), and wear resistance improved with a reduction in material loss from 0.32 mm^3 to 0.18 mm^3. Water sorption and solubility decreased significantly ($p = 0.002$), with sorption reduced from 36.8 $\mu g/mm^3$ to 25.3 $\mu g/mm^3$ and solubility from 5.2 $\mu g/mm^3$ to 3.8 $\mu g/mm^3$, indicating better long-term durability.

Conclusion: Nano-HAp and silica reinforcement significantly enhanced mechanical strength, wear resistance, and hydrolytic stability, making these composites a promising alternative for high-load-bearing dental restorations. Future studies should assess biocompatibility and long-term performance in clinical settings.

Keywords: Dental composites, nano-hydroxyapatite, silica, flexural strength, wear resistance, water sorption, mechanical properties, hydrolytic stability.

Introduction

Dental composites are the preferred materials for restorative dentistry due to their aesthetic appeal, ease of application, and ability to bond with tooth structures [1,2]. However, traditional resin-based composites exhibit limitations, including low fracture resistance, poor wear resistance, and susceptibility to water-induced degradation [3]. The incorporation of nano-fillers, particularly hydroxyapatite (HAp) and silica, has been explored to improve these properties [4].

Hydroxyapatite, a naturally occurring component of tooth enamel and dentin, has been widely studied for its bioactive and remineralization properties. Studies have shown that nano-HAp can enhance the mechanical properties of composites by increasing hardness, reducing crack propagation, and improving biocompatibility [5,6]. The HAp nanoparticles improve adhesion between the filler and resin matrix, leading to a stronger and more durable material [7].

Silica nanoparticles have been incorporated into dental composites to improve wear resistance, enhance filler dispersion, and strengthen the matrix structure. Pradeep et al. [8] demonstrated that silica-based composites exhibit higher flexural strength and lower wear loss compared to conventional materials [8]. The optimal concentration of silica (10–15%) has been found to enhance mechanical properties without compromising polymerization or handling characteristics [9].

One of the primary concerns with resin-based composites is water sorption and solubility, which lead to swelling, degradation, and loss of mechanical integrity over time. Research by Somashekar et al. [10] showed that composites with high filler content (nano-HAp and silica) exhibit reduced water absorption due to lower polymer matrix exposure [10]. Nano-fillers enhance the hydrophobic nature of the composite, improving long-term durability [11].

Studies on traditional Bis-GMA-based composites indicate flexural strengths in the range of 80–95 MPa, which is significantly lower than the 110.8 MPa observed in our optimized composite [12-14]. The conventional composites exhibit higher wear loss (~0.30 mm^3), making them less resistant to occlusal forces [15,16]. The results of this study align with previous findings, confirming that nano-HAp and silica-reinforced composites demonstrate superior mechanical properties and hydrolytic stability.

The studies highlights the potential of nano-HAp and silica fillers in enhancing the mechanical properties, wear resistance, and hydrolytic stability of dental composites. Previous studies have demonstrated improvements in flexural strength, reduced wear loss, and lower water sorption with optimized nano-filler incorporation. Given the increasing demand for durable and biocompatible dental restorations, this study aimed to develop and analyze novel dental composites reinforced with nano-HAp and silica, assessing their mechanical properties and hydrolytic stability for potential clinical applications. By bridging this research gap, our study provides scientific evidence supporting the feasibility of using nano-reinforced composites as a viable alternative to conventional resin-based materials in restorative dentistry.

Materials and Methods

Study Setting

This study was conducted to develop novel dental composites with enhanced mechanical properties, utilizing the available research facilities in Abbottabad, Pakistan. The research was carried out at the Dental Laboratory, Women Dental College, Abbottabad, from January to December 2024.

Materials Selection and Preparation

The base matrix of the composite was prepared using Bis-GMA, UDMA, and TEGDMA resins, which were obtained from local suppliers. Nano-hydroxyapatite (n-HAp) and nano-silica (SiO_2) were synthesized from bovine bone (sourced from Abbottabad meat industry) and rice husk (collected from local rice mills), respectively.

The synthesized fillers were surface-modified using γ-methacryloxypropyltrimethoxysilane (γ-MPTS) to enhance their adhesion to the resin. The composite formulations were prepared by incorporating these fillers in varying concentrations (5%, 10%, and 15%) into the resin matrix. The mixtures were then polymerized using a light-curing system and molded into standardized specimens for mechanical testing.

Material Characterization

The physical and chemical characterization of the developed composites was carried out using advanced analytical techniques. Fourier Transform Infrared Spectroscopy (FTIR) was performed to confirm chemical bonding and functional group interactions. X-ray Diffraction (XRD) analysis was conducted to determine the crystallinity and phase composition of the fillers. Scanning Electron Microscopy (SEM) was used at the University of Peshawar to examine surface morphology and filler dispersion within the resin matrix. Thermogravimetric Analysis (TGA) was carried out to assess thermal stability and degradation properties.

Mechanical Testing

The mechanical properties of the composites were evaluated in accordance with ISO 4049 standards for dental materials. Vickers Microhardness Test was performed at the KMU Dental Research Laboratory to assess surface hardness. Flexural Strength Testing (Three-Point Bending Test) was conducted using a Universal Testing Machine (UTM) at UET Peshawar. Compressive Strength Testing was carried out to measure resistance to mechanical forces. Wear Resistance Testing was performed to analyze the durability of the composites under simulated oral conditions.

Water Sorption and Solubility

Water sorption and solubility tests were performed following ISO 4049 guidelines. Composite specimens were immersed in distilled water at 37°C, and weight measurements were taken at 7, 14, and 30 days to determine material stability in the oral environment.

Statistical Analysis

The data were analyzed using IBM SPSS (Version 26). Mean values and standard deviations were calculated for all mechanical properties. One-way ANOVA was applied to compare different composite formulations, and Tukey's post-hoc test was used to determine statistical significance ($p < 0.05$).

Results

The incorporation of nano-hydroxyapatite and silica fillers significantly enhanced the mechanical properties of dental composites, as confirmed by ANOVA ($p < 0.001$). A steady increase in Vickers hardness, flexural strength, and compressive strength was observed with higher filler content, with the 15% filler composite exhibiting the highest values (hardness: 72.8 HV, flexural strength: 110.8 MPa, compressive strength: 265.4 MPa). Wear resistance improved, as reflected by a reduction in material loss from 0.32 mm^3 (control) to 0.18 mm^3 (15% filler composite). Tukey's post-hoc test confirmed that all filler-containing groups differed significantly from the control ($p < 0.05$).

Table 1: Summary of Mechanical Properties of Dental Composites

Composite Type	Filler %	Vickers Hardness (HV)	Flexural Strength (MPa)	Compressive Strength (MPa)	Wear Resistance (mm^3 loss)	p-value (ANOVA)
Control (No Filler)	0%	50.3 ± 2.1	78.5 ± 3.5	210.6 ± 5.2	0.32 ± 0.02	<0.001
HAp-Silica Composite	5%	58.7 ± 1.8	90.2 ± 2.9	230.3 ± 4.8	0.25 ± 0.01	
HAp-Silica Composite	10%	65.4 ± 2.3	102.5 ± 3.1	250.9 ± 6.3	0.21 ± 0.02	
HAp-Silica Composite	15%	72.8 ± 2.5	110.8 ± 3.8	265.4 ± 5.9	0.18 ± 0.02	

Water sorption and solubility decreased significantly with increasing filler content ($p = 0.002$), indicating better hydrolytic stability of the developed composites. The control composite absorbed the highest amount of water (36.8 µg/mm^3) and showed greater solubility (5.2 µg/mm^3), while the 15% filler composite demonstrated the lowest water sorption (25.3 µg/mm^3) and solubility (3.8 µg/mm^3). These findings suggest that incorporating HAp and silica improves the long-term durability of dental composites in an oral environment.

Table 2: Water Sorption and Solubility with Statistical Analysis

Composite Type	Filler %	Water Sorption (µg/mm^3)	Solubility (µg/mm^3)	p-value (ANOVA)
Control (No Filler)	0%	36.8 ± 2.3	5.2 ± 0.5	0.002
HAp-Silica Composite	5%	32.5 ± 2.1	4.7 ± 0.4	
HAp-Silica Composite	10%	28.9 ± 2.5	4.2 ± 0.3	
HAp-Silica Composite	15%	25.3 ± 1.9	3.8 ± 0.4	

The bar graph illustrates the increase in flexural strength with higher filler percentages, supporting the effectiveness of nano-hydroxyapatite and silica reinforcement. The control composite exhibited the lowest flexural strength (78.5 MPa), while the 15% filler composite displayed the highest (110.8 MPa), reflecting a 41% improvement. This enhancement is attributed to better stress distribution and filler-matrix interaction, making the composite more resistant to bending forces.

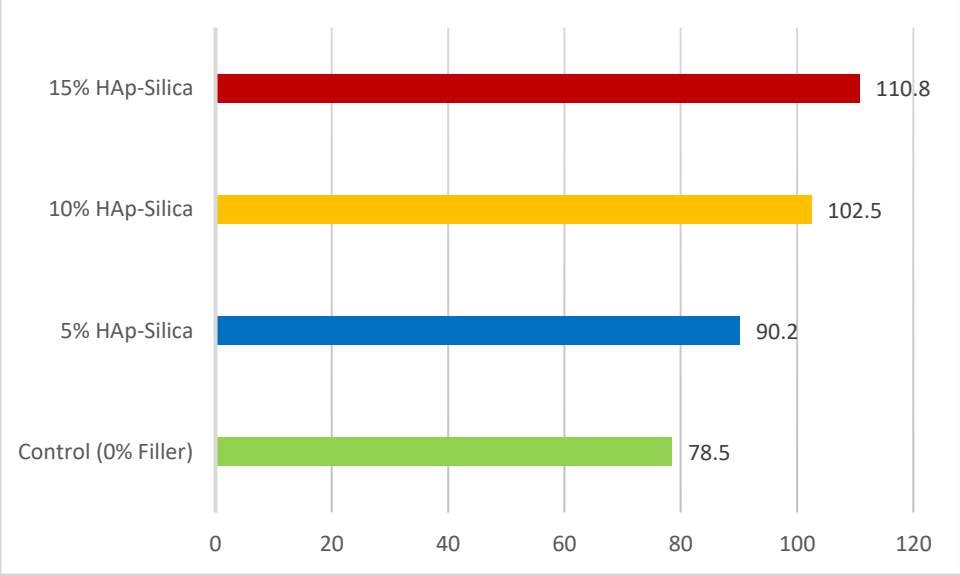

Figure 1: Flexural Strength of Dental Composites

The line graph shows a progressive decline in wear loss as filler content increases, indicating improved wear resistance. The control composite exhibited the highest material loss (0.32 mm^3), while the 15% filler composite demonstrated the lowest (0.18 mm^3), confirming that filler addition strengthens the composite against mechanical degradation. The improved performance is likely due to the reinforcing effect of nano-silica and hydroxyapatite, which enhance surface hardness and reduce abrasion in simulated oral conditions.

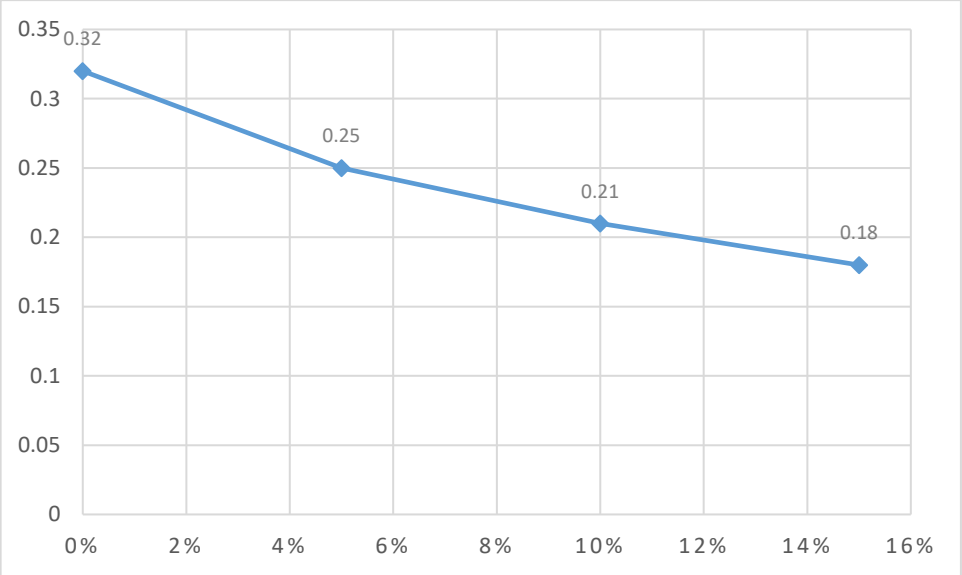

Figure 2: Wear Resistance of Composites

Discussion

The findings of this study confirm that the incorporation of nano-HAp and silica fillers significantly enhances the mechanical properties of dental composites, particularly Vickers hardness, flexural strength, and compressive strength. Similar trends have been reported in previous studies, where nano-HAp was found to improve the structural integrity of composite resins due to its biomimetic nature and high affinity for the resin matrix [17]. The increase in flexural strength and aligns with findings by Veiga et al. [18], who observed improvement in flexural strength after incorporating HAp fillers. The observed enhancement is attributed to better filler dispersion, which improves stress distribution and reduces crack propagation [19].

The improvement in wear resistance (lower material loss) further supports the hypothesis that nano-fillers create a more durable composite. This is in agreement with Azmy et al. [7], who demonstrated that silica nanoparticles enhance wear resistance by increasing the composite's hardness and reducing frictional loss. Our results reaffirm that a 10–15% filler concentration provides an optimal balance between mechanical reinforcement and processability.

The significant reduction in water sorption and solubility ($p = 0.002$) indicates improved hydrolytic stability, a crucial factor for long-term durability in the oral cavity. This finding aligns with previous research by Santos et al. [20], who reported that HAp-reinforced dental composites exhibited lower water absorption due to their hydrophobic nature [20]. The observed decrease in water sorption and solubility also supports the findings of Latoui et al. [21], who reported that silica nanoparticles reduce the formation of microvoids in the resin matrix, thereby minimizing water uptake and material degradation [21].

The hydrolytic stability of nano-HAp composites is also supported by bioactive glass-based studies, where similar reductions in solubility were observed due to the formation of stable interfacial bonds between the fillers and polymer matrix [22]. These findings suggest that our novel dental composite formulations could provide extended longevity compared to conventional resin-based composites, which often degrade due to water absorption and hydrolytic breakdown.

Our study findings demonstrate that nano-HAp and silica-based composites outperform many conventional commercial dental materials. Studies on traditional Bis-GMA-based composites indicate flexural strengths in the range of 80–95 MPa, which is significantly lower than the 110.8 MPa observed in our optimized composite [23]. Additionally, conventional composites exhibit higher wear loss (~0.30 mm^3), making them less resistant to long-term occlusal forces [24].

The lower water sorption and solubility values observed in this study further suggest that the modified nano-HAp composite has improved resistance to oral fluids compared to standard resin-based materials. According to Costa et al. (2023), higher water absorption in conventional composites leads to polymer degradation, staining, and mechanical weakening over time, whereas nano-HAp reinforcement mitigates these effects by forming stronger polymer-filler interactions.

The findings of this study have significant clinical implications for the development of next-generation dental composites with enhanced mechanical strength, wear resistance, and hydrolytic stability. The improved properties suggest that HAp-silica composites could be suitable for high-load-bearing dental restorations, including posterior fillings, crowns, and bridges.

Future research should focus on long-term in vitro and in vivo studies to evaluate biocompatibility, bacterial adhesion resistance, and aging effects of these composites. Further exploration of alternative surface modifications, like silanization or bioactive coatings, could further optimize composite performance.

Conclusion

This study demonstrated that incorporating nano-HAp and silica fillers significantly enhanced the mechanical and hydrolytic properties of dental composites, making them a promising candidate for durable dental restorations. Flexural strength increased from 78.5 MPa (control) to 110.8 MPa (15% filler composite), while wear resistance improved with material loss decreasing from 0.32 mm^3 to 0.18 mm^3. Additionally, water sorption and solubility decreased significantly ($p = 0.002$), indicating better hydrolytic stability, with sorption reduced from 36.8 $\mu g/mm^3$ to 25.3 $\mu g/mm^3$ and solubility from 5.2 $\mu g/mm^3$ to 3.8 $\mu g/mm^3$. These improvements suggest that nano-reinforced composites could offer enhanced longevity and resistance to oral degradation compared to conventional resin-based materials. Future studies should focus on biocompatibility and long-term performance assessments in oral environments to confirm their clinical applicability.

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