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Effect of 5 wt.% Nanohydroxyapatite Reinforcement on the Surface Microhardness of Nanohybrid and Microhybrid Resin Composites: An in vitro comparative Study

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KEYWORDS

Composite resins, microhybrid composite, nanohybrid composite, nanohydroxyapatite, nanotechnology.

ABSTRACT:

Introduction: Advancements in restorative dentistry have increased the use of microhybrid and nanohybrid composites due to their strength and esthetics. The addition of nanohydroxyapatite (nHA) shows potential to further improve mechanical properties and bioactivity. Microhardness, which reflects resistance to wear, is crucial for restoration longevity; however, limited evidence exists comparing the effect of nHA on microhybrid and nanohybrid composites.

Objectives To evaluate and compare the surface microhardness of nanohybrid and microhybrid resin composites, both unmodified and modified with 5 wt% nanohydroxyapatite.

Methods: An in vitro experimental study was conducted on 20 samples, divided into four groups (n = 5): unmodified nanohybrid composite, nanohybrid composite modified with 5 wt% nHA, unmodified microhybrid composite, and microhybrid composite modified with 5 wt% nHA. Specimens were prepared using standardized Teflon molds and polymerized using a light-curing unit. Modified composites were obtained by incorporating 5 wt% nHA through a combination of manual and mechanical mixing. Following storage at 37°C for 24 hours, surface microhardness was assessed using a Vickers hardness tester. Statistical analysis was performed using one-way ANOVA followed by Tukey's post hoc test ($\alpha = 0.05$).

Results: A statistically significant difference in Vickers hardness was observed among the groups ($p < 0.001$), with nanohybrid composites demonstrating higher hardness values compared to microhybrid composites. The incorporation of 5 wt% nHA did not result in a statistically significant change in microhardness within the same composite category.

Conclusions: Nanohybrid composites exhibited superior surface microhardness compared to microhybrid composites. The type of composite material had a greater influence on hardness than the addition of nanohydroxyapatite.

1. Introduction

Advancements in restorative dentistry have significantly improved the materials used to restore tooth structure, with composite resins becoming a preferred choice due to their aesthetics and enhanced mechanical properties[1]. Among these, microhybrid and nanohybrid composites are commonly used because they offer a good balance of strength, wear resistance, and polishability[2].Microhybrid composites contain a mix of fine and microfill particles, providing adequate strength and durability, though they may fall short in long-term polish retention and esthetics[3]. In comparison, nanohybrid composites incorporate nanosized fillers, which improve surface smoothness, translucency, and overall mechanical performance[4]. This evolution toward nanotechnology has played a key role in developing materials with better clinical outcomes. Hydroxyapatite (HA), a calcium phosphate compound similar to the mineral component of enamel and bone, is known for its biocompatibility and remineralization potential[5]. Its nanoscale form, nanohydroxyapatite (nHA), offers an even higher surface area and improved biological and mechanical properties[6]. When added to resin composites, nHA can enhance properties such as microhardness and wear resistance while also supporting remineralization[7].

Microhardness is an important factor in determining a material's resistance to surface wear and its longevity under functional forces[8]. Although both microhybrid and

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nanohybrid composites have been studied individually, there is limited research comparing the effect of nHA modification on their microhardness. Understanding this can help in selecting the most suitable material for improved clinical performance[9].

2. Objectives

To evaluate and compare the surface microhardness of nanohybrid and microhybrid resin composites, both unmodified and modified with 5 wt% nanohydroxyapatite.

3. Methods

A total sample size of 20 specimens was determined, with 5 samples in each of the four groups. Acrylic resin blocks were fabricated using a square Teflon mold measuring 35 mm in height and 25 mm in diameter. A standardized cylindrical cavity (4 mm diameter × 2 mm depth) was prepared at the center of each block using a 330 carbide bur, and all dimensions were verified with a digital vernier caliper.

Modified composites were prepared by incorporating 5 wt% nanohydroxyapatite (nHA) into the resin composite (0.05 g nHA with 0.95 g composite). Initially, manual pre-mixing was performed using a plastic spatula to ensure uniform blending and to reduce particle agglomeration. This was followed by mechanical mixing in a speed mixer (dual asymmetric centrifuge) at 2000–3000 rpm for 1–3 minutes in a sealed container to achieve optimal dispersion. The final mixture was then transferred into syringes or molds and stored in a light-proof container to prevent premature curing.

The samples were randomly divided into four groups (n = 5):

- Group 1: Nanohybrid composite
- Group 2: Nanohybrid composite modified with 5 wt% nHA
- Group 3: Microhybrid composite
- Group 4: Microhybrid composite modified with 5 wt% nHA

The composite material was placed into the prepared cavities using a Teflon mold (4 mm diameter × 2 mm height) in a single increment. Group 1 and Group 2 used bulk-fill composite (3M ESPE), while Group 3 and Group 4 used packable composite (Dentsply Spectrum). All samples were light-cured for 20 seconds using a curing unit with a wavelength range of 420–480 nm and an irradiance of 1200 mW/cm². Excess material was removed, and the samples were then stored in a humidor at 37°C and 100% humidity for 24 hours. All procedures were carried out by a single operator to maintain consistency.

Microhardness testing was performed using a Vickers hardness tester. Each specimen was properly positioned, and a load was applied to the surface using an indenter oriented parallel to the surface at a crosshead speed of 0.5 mm/min. The maximum force causing indentation was recorded, and the obtained data were subjected to statistical analysis.

4. Results

The mean Vickers hardness number (VHN) varied significantly among the four groups. Group 2 (nanohybrid composite + 5% nanohybrid) demonstrated the highest mean VHN (99.2 ± 1.73), followed by Group 1 (nanohybrid composite) with a mean of 93.7 ± 2.1 (Table 1). In contrast, lower hardness values were observed in Group 4 (microhybrid + 5% nanohybrid; 77.26 ± 5.23) and Group 3 (microhybrid composite; 75.4 ± 4.65)

One-way ANOVA revealed a statistically significant difference in VHN among the groups ($F = 49.640$; $p < 0.001$) Tukey's post-hoc analysis showed no significant difference between Group 1 and Group 2 ($p = 0.145$), and between Group 3 and Group 4 ($p = 0.876$) (Table 2). However, both nanohybrid groups (Groups 1 and 2) exhibited significantly higher hardness values compared to the microhybrid groups (Groups 3 and 4) ($p < 0.001$) (Table 3).

Table 1: Descriptive statistics of Vickers hardness number between three study groups

	Mean	SD	SE	Min.	Max.
Group 1 (Nanohybrid composite)	**93.7**	**2.1**	**0.94**	**91.4**	**96.5**
Group 2	**99.2**	**1.73**	**0.77**	**97.0**	**101.3**

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(Nanohybrid + 5% Nanohybrid)					
Group 3 (Micro hybrid)	**75.4**	**4.65**	**2.08**	**70.0**	**81.0**
Group 4 (Micro hybrid + 5% Nanohybrid)	**77.26**	**5.23**	**2.34**	**70.5**	**83.6**

Table 2: Comparative statistics of Vickers hardness number between three study groups using One way Anova F test

	Mean	SD	One-way Anova F test	P value, Significance
Group 1 (Nanohybrid composite)	**93.7**	**2.1**	**F = 49.640**	**p<0.001****
Group 2 (Nanohybrid + 5% Nanohybrid)	**99.2**	**1.73**		
Group 3 (Micro hybrid)	**75.4**	**4.65**		
Group 4 (Micro hybrid + 5% Nanohybrid)	**77.26**	**5.23**		

Table 3: Pairwise comparative statistics of Vickers hardness number between three study groups using Tukey's post hoc test

Group	Comparison	Mean Difference	P value, Significance
Group 1 (Nanohybrid composite) vs	**Group 2 (Nanohybrid + 5% Nanohybrid)**	**5.42**	**p=0.145 (NS)**
	Group 3 (Micro hybrid)	**18.3**	**p<0.001****
	Group 4 (Micro hybrid + 5% Nanohybrid)	**16.52**	**p<0.001****
Group 2 (Nanohybrid + 5% Nanohybrid) vs	**Group 3 (Micro hybrid)**	**23.72**	**p<0.001****
	Group 4 (Micro hybrid + 5% Nanohybrid)	**21.94**	**p<0.001****

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Group 3 (Micro hybrid) vs	**Group 4 (Micro hybrid + 5% Nanohybrid)**	**1.78**	**p=0.876 (NS)**

5. Discussion

Resin-based composites are complex materials composed of an organic resin matrix reinforced with inorganic filler particles, where the size, distribution, morphology, and interfacial bonding of fillers play a critical role in determining mechanical properties such as surface microhardness, wear resistance, and durability[1,2]. Microhybrid composites were originally developed to achieve higher filler loading and improved strength; however, their relatively larger particle size may limit polishability and long-term surface smoothness[3]. In contrast, nanohybrid composites incorporate nanoscale fillers, which enhance filler packing density, reduce interparticle spacing, and improve filler–matrix interaction, ultimately leading to superior mechanical and esthetic properties[4,5].

The present study demonstrated that nanohybrid composites exhibited significantly higher surface microhardness compared to microhybrid composites. This finding agrees with several previous studies, which reported that nanosized fillers improve stress distribution within the resin matrix and increase resistance to surface indentation[6,7]. Beun et al.[2] reported that nanofilled and nanohybrid composites showed superior hardness due to improved filler dispersion and enhanced interfacial bonding between filler particles and resin matrix. Similarly, Ilie and Hickel[3] attributed increased hardness in nanocomposites to higher polymer cross-link density and more efficient load transfer across the filler–matrix interface. Additional studies have also confirmed that nanohybrid composites demonstrate improved mechanical behavior under varying curing conditions and loading stresses[8,9].

The improved performance of nanohybrid composites can also be explained by the increased surface area of nanoparticles, which allows better silane coupling and stronger adhesion between the filler and the resin matrix[10]. This enhanced bonding reduces the likelihood of filler debonding and microcrack formation under functional stresses[11]. Moreover, the uniform distribution of nanoparticles contributes to reduced polymerization shrinkage stress and improved structural integrity[12].

Nanohydroxyapatite (nHA), owing to its chemical similarity to natural enamel and dentin, has been widely investigated as a bioactive filler in restorative dentistry[13]. It has the potential to enhance remineralization, promote ion exchange, and improve the biological properties of composite materials[14]. However, in the present study, incorporation of 5 wt% nHA did not produce a statistically significant increase in surface microhardness within either composite group. This observation is consistent with findings from previous investigations, which suggest that the reinforcing ability of nHA is highly dependent on its concentration, dispersion, and interaction with other fillers[15].

At lower concentrations, such as 5 wt%, nHA particles may not contribute significantly to mechanical reinforcement due to possible agglomeration or inadequate stress transfer within the matrix[16]. Mirajkar et al.[5] reported that while nHA improves bioactivity, its effect on hardness is limited unless combined with other reinforcing fillers or used at higher concentrations. Similarly, other studies have indicated that excessive or poorly dispersed nanoparticles may even act as stress concentrators, negatively affecting mechanical properties[17].

The lack of significant improvement in microhardness in this study suggests that nHA, at the tested concentration, functions primarily as a biofunctional additive rather than a mechanical enhancer. Importantly, its incorporation did not adversely affect the hardness of the composites, which is clinically relevant[18]. This indicates that bioactive modifications can be introduced without compromising essential mechanical properties.

From a clinical perspective, surface microhardness is directly related to wear resistance and longevity of restorations under masticatory forces[19]. The superior hardness of nanohybrid composites observed in this study supports their use in stress-bearing areas. Additionally, the incorporation of nHA may offer added benefits such as remineralization potential and improved biocompatibility, even if it does not significantly enhance hardness at lower concentrations[20].

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Limitations of the study: -

However, this study has certain limitations. Being an in vitro study, it does not fully replicate the complex oral environment, where factors such as thermal cycling, pH variations, and mechanical fatigue may influence material performance. Future studies should evaluate different concentrations of nHA, long-term aging effects, and other mechanical properties such as flexural strength, fracture toughness, and wear resistance to provide a more comprehensive understanding.

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