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Original Research Article

The effect of a 16% carbamide peroxide on the microhardness and compressive strength of a nano composite

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Abstract

Aim: This in vitro study was done to evaluate the effect of a 16% carbamide peroxide bleaching agent on the micro hardness and compressive strength of a Nanofilled Resin composite restorative material.

Materials and Methods: 60 cylindrical shaped specimens (5×7mm) of a nanofilled resin composite material were fabricated and then polished with fine polishing disc. After being polished, specimens were cleaned with distilled water for 2 minutes to remove any surface debris and then stored in distilled water for 24 hours. Specimens were divided into three groups (n=20). One group was selected as a control group (non-treated with bleaching agent). The other two groups were treated with bleaching agent for 14 days (group A) and for 14 days followed by immersion in artificial saliva for 14 days (group B). The microhardness of the specimens was measured using the vickers hardness test and compressive strength using universal testing machine.

Result: In this study, the microhardness and compressive strength of the control group were greater than those of Group A, which in turn were greater than or equal to those of Group B.

Conclusion: Bleaching has an adverse effect on compressive strength and microhardness of nanofilled composite.

Keywords: Nano filled composite resin, Carbamide peroxide, Composite resin hardness, Visible light cure unit

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1. Introduction

The appearance of teeth plays a significant role in a person's overall look. Several factors influence dental aesthetics, including tooth color, shape, alignment, and the quality of dental restorations. Among these, tooth color is a key factor in determining an individual's satisfaction with their smile. Vital tooth bleaching involves the application of a chemical solution to the tooth surface to achieve a whitening effect.[1] All tooth whitening treatments utilize either hydrogen peroxide or carbamide peroxide. Carbamide peroxide is commonly used as a home bleaching agent. The benefits of home bleaching include its ease of application, affordability, and high success rate. However, the use of chemicals in teeth whitening remains a topic of debate, as it may have potential effects on both hard and soft oral tissues. Using teeth whitening agents in high concentrations or for extended periods may lead to excessive tooth sensitivity and potential damage to the mucosa and gingiva. The negative effects of teeth whitening are often linked to factors such as low pH, oxidation reactions, and the chemical composition of whitening agents. Several factors can influence the teeth whitening process, including the type, concentration, and duration of application of the whitening material, as well as light and temperature. Other contributing factors include the type of discoloration, the original tooth color before whitening, and the presence of plaque or calculus, which may hinder the whitening process. The pH level of the whitening material also plays a role, as it varies depending on the bleaching agent used. A low pH can lead to enamel erosion. Additionally, the duration of contact between the whitening material and the tooth surface affects the results—the longer the exposure, the more pronounced the whitening effect. Patients seeking teeth whitening may have metal-based,

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resin-based, or other types of restorations in their anterior or posterior teeth. The chemical processes involved in bleaching agents can potentially impact the clinical durability of tooth-colored restorations. Bleaching agents can affect dental restorative materials by altering their surface morphology as well as their chemical and physical properties.[2] One of the key physical properties of restorative materials is surface hardness, which refers to their resistance to indentation or penetration. Research on the effects of bleaching agents on the microhardness of resin-based restorative materials has yielded conflicting results. While some studies have reported a decrease in surface microhardness after bleaching, others have found no significant changes.[3]

Therefore, this in vitro study was undertaken to evaluate the effect of a 16% carbamide peroxide on the microhardness and compressive strength of a nano composite.

2. Materials and Methods

2.1. Specimen fabrication

Tetric N-Ceram A2 shade was used as a Nano filled composite resin restorative material in the current study and its compositions, with the home bleaching agent, are shown in **(Table 1)**.

60 Cylindrical specimens (5.0 mm diameter × 7.0 mm depth) were fabricated in shade A2. Cylindrical rubber molds **(Figure 1)** were positioned on a transparent plastic matrix strip lying on a glass plate. The composite resin restorative materials were placed in 2.0-mm increments. After placing the materials into the mold, a transparent plastic matrix strip was positioned over them **(Figure 2)**, and a glass plate was secured to create a smooth surface. Each specimen underwent light polymerization for 40 seconds using a visible light curing unit (ivoclar vivadent) with an intensity range of 480 to 520 mW/cm². **(Figure 3)** The specimens were then polished using medium, fine, and superfine polishing discs (Sof-Lex, 3M ESPE) on a slow-speed handpiece rotating in a single direction. A micrometer (Ultra-Cal Mark III, Fowler Tools and Instruments, Sylvac, Newton, MA, USA) was used to verify the final thickness of the polished specimens. Following polishing, the specimens underwent ultrasonic cleaning with distilled water for 2minutes to eliminate any surface debris. Finally, all specimens were stored in distilled water at 37°C for 24 hours.

Chief materials are listed in **(Figure 4)**. Specimens **(Figure 5)** from each material were divided randomly into three groups (20/group): **(Figure 6)**

1. **Control group:** Immersed for 14 days in artificial saliva, with no bleaching treatment
2. **Group A:** Treated with a 16% carbamide peroxide bleaching agent for 14 days
3. **Group B:** Treated with a 16% carbamide peroxide bleaching agent for 14 days and then immersed in artificial saliva for 14 days

Figure 1: Cylindrical rubber mold

Figure 2: Preparation of specimen

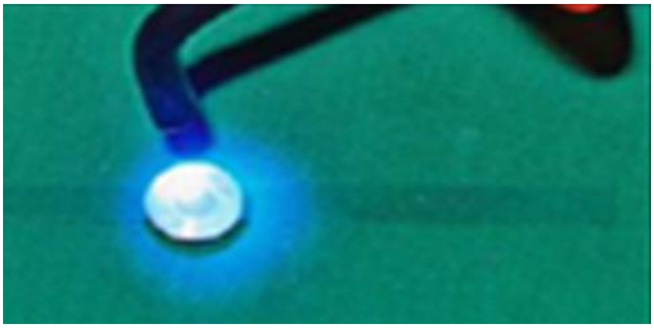


Figure 3: Light curing of composite resin specimen

2.2. Control group

The specimens in the control group were stored in artificial saliva for 14 days at 37°C and no bleaching, followed by immersion in distilled water for 24 h at 37°C in preparation for the microhardness test. The artificial saliva was replaced daily.

Figure 4: Chief materials

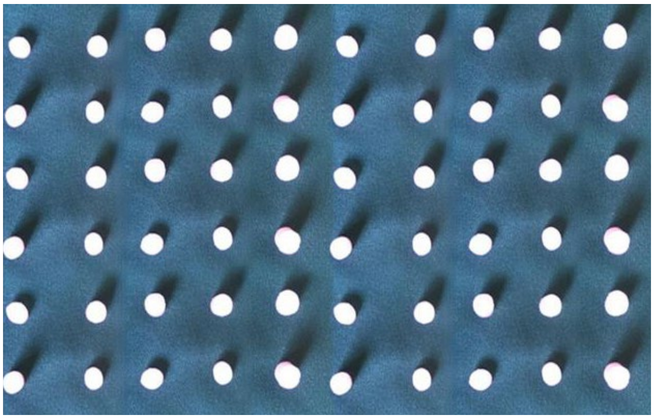


Figure 5: Total specimens

2.3. Bleaching procedure

The bleaching procedure was performed on the top surfaces of the specimens in groups A and B, with an at-home bleaching material 16% carbamide peroxide. The bleaching agent covered the top surfaces of the specimens at a thickness of 1.0 mm. At the end of every bleaching application, the treated specimens were washed, first with a soft toothbrush under flowing distilled water and then in an ultrasonic cleaner for 5 minutes. They were then placed in fresh artificial saliva for 16 hours at 37° C until the next application. The artificial saliva was replaced daily. In groups A and B, bleaching gel was applied for 8 hours daily for 14 consecutive days at 37° C, to simulate at-home bleaching. In group A, after the 14-day bleaching procedure, all specimens were stored in distilled water for 24 hours at 37° C in preparation for the microhardness and compressive strength test. In group B, after the 14-day bleaching procedure, all specimens were stored in artificial saliva for 14 days at 37° C, followed by immersion in distilled water for 24 hours at 37° C, in preparation for the microhardness and compressive strength test.

Figure 6: All 3 groups immersed in distilled water for 24 hours

2.4. Vickers hardness test and Compressive strength test

The micro hardness value of each samples were tested using Vicker's micro hardness tester on top. The specimens were placed on the platform, with the surface being tested facing the diamond indenter. A load of 300 g was applied to the surface for a 15 s dwell time **(Figure 7)**. The compressive strength of the samples were tested using universal testing machine **(Figure 8)**.

Figure 7: Vickers hardness test

Figure 8: Compressive strength test

2.5. Statistical analysis

Statistical calculations were performed with SPSS version 16.0 software (SPSS Inc., Chicago, IL, USA). Normality of the data was assessed using Shapiro Wilkinson test. Inferential statistics to find out the difference between the groups was done using One-Way ANOVA TEST followed by Boneferroni Posthoc test.

3. Results

Results of the study shown in **(Table 2)**

Table 1: Composition of nanofilled composite resin Tetric N-Ceram

Tetric N – Ceram (Ivoclar vivadent AG)		
Matrix	**Isofiller**	**Loading**
Bis GMA	Ytterbium fluoride	75-77% by weight (53-55% by volume)
Bis EMA	Mixed oxides	
UDMA	Additives	
Standard filler Ba-Al-Si	Catalyst	
Glass with 2 mean filler	Stabilizer	
Size	Pigments	

Table 2: Test results

Compressive strength			
Sample	**Control**	**Group A**	**Group B**
1	265.18	263.2	263.15
2	265.2	260.31	263.25
3	264.17	261.32	258.1
4	265.15	260.25	261.2
5	266.21	259.18	259.18
6	264.22	261.15	262.14
7	264.21	258.23	260.12
8	267.2	260.15	261.13
9	266.21	258.25	259.15
10	265.22	260.23	261.11
Microhardness			
Sample	**Control**	**Group A**	**Group B**
1	79.2	80.18	80.18
2	90.57	81.02	81.01
3	90.77	79.53	79.43
4	88.37	79.63	80.55
5	88.1	80.67	80.27
6	87.2	80.77	80.97
7	88.85	78.83	79.81
8	85.37	78.23	78.22
9	82.27	79.3	79.35

Table 3: Descriptive statistics

	Descriptive statistics						
		N	**Minimum**	**Maximum**	**Mean**		**Std. deviation**
		Statistic	**Statistic**	**Statistic**	**Statistic**	**Std. error**	**Statistic**
Compressive Strength	Control	10	264.17	267.20	265.2970	.31536	.99726
	Group A	10	258.23	263.20	260.2270	.47012	1.48666
	Group B	10	258.10	263.25	260.8530	.54525	1.72423
Micro Hardness	Control	10	79.20	90.77	86.4200	1.19379	3.77511
	Group A	10	78.23	81.02	79.7360	.28688	.90721
	Group B	10	78.22	82.01	79.9980	.33328	1.05392

Table 4: One way ANOVA test of Compressive strength

ANOVA					
Compressive strength					
	Sum of squares	df	Mean square	F	Sig.
Between Groups	152.820	2	76.410	37.106	.000
Within Groups	55.599	27	2.059		
Total	208.419	29			

Table 5: Boneferroni Posthoc test of Compressive strength

Multiple comparisons						
Dependent Variable: Compressive strength						
Bonferroni						
(I)	(J)	Mean difference (I-J)	Std. error	Sig.	95% Confidence Interval	
					Lower bound	Upper bound
Control	Group A	5.07000*	.64175	.000	3.4320	6.7080
	Group B	4.44400*	.64175	.000	2.8060	6.0820
Group A	Control	–5.07000*	.64175	.000	–6.7080	–3.4320
	Group B	–.62600	.64175	1.000	–2.2640	1.0120
Group B	Control	–4.44400*	.64175	.000	–6.0820	–2.8060
	Group A	.62600	.64175	1.000	–1.0120	2.2640

*. The mean difference is significant at the 0.05 level.

Table 6: One way ANOVA test of Microhardness

ANOVA					
Microhardness					
	Sum of squares	df	Mean square	F	Sig.
Between Groups	286.622	2	143.311	26.563	.000
Within Groups	145.667	27	5.395		
Total	432.289	29			

Table 7: Boneferroni posthoc test of microhardness

Multiple comparisons						
Dependent variable: Microhardness						
Bonferroni						
(I)	(J)	Mean difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower bound	Upper bound
CONTROL	Group A	6.68400*	1.03876	.000	4.0326	9.3354
	Group B	6.42200*	1.03876	.000	3.7706	9.0734
GROUP A	Control	–6.68400*	1.03876	.000	-9.3354	-4.0326
	Group B	–.26200	1.03876	1.000	-2.9134	2.3894
GROUP B	Control	–6.42200*	1.03876	.000	-9.0734	-3.7706
	Group a	.26200	1.03876	1.000	-2.3894	2.9134

*. The mean difference is significant at the 0.05 level.

Based on the descriptive data **(Table 3)** The control group showed superior performance in both mechanical parameters. The application of 16% carbamide peroxide over 14 days clearly compromised the integrity of the nanofilled composite resin. Post-bleaching immersion in artificial saliva (Group B) did not significantly improve either compressive strength or microhardness. These findings align with the study's broader conclusion that bleaching agents, even at-home concentrations, may negatively affect the clinical durability of composite restorations.

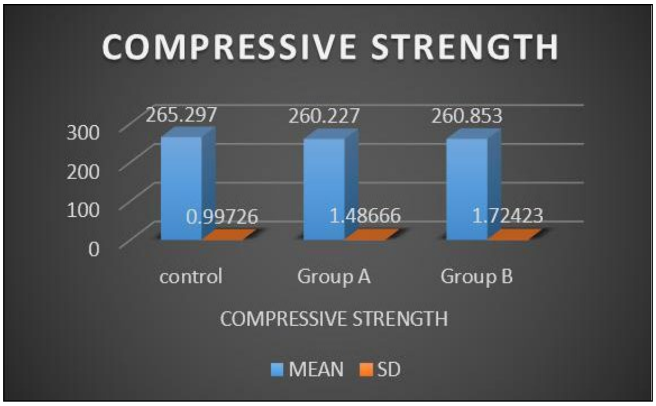
The ANOVA results **(Table 4)** showed a highly significant difference (p = 0.000) in compressive strength between the groups.The F-value of 37.106 indicates a substantial variance in compressive strength attributable to the treatment condition rather than random variation.These findings suggest that bleaching with 16% carbamide peroxide had a statistically significant negative impact on the compressive strength of the nanocomposite material.Post-bleaching treatment with artificial saliva (Group B) did not restore the compressive strength to control levels.

The Bonferroni post hoc test **(Table 5)** was conducted following a significant ANOVA result to identify which pairs of groups differed significantly in terms of compressive strength. Control vs Group A and Control vs Group B showed statistically significant differences (p = 0.000), indicating that both bleaching protocols significantly reduced compressive strength compared to the unbleached control. Group A vs Group B showed no significant difference (p = 1.000), suggesting that post-bleaching immersion in artificial saliva did not restore the compressive strength.

A one-way ANOVA test was conducted to compare the microhardness **(Table 6)** values among the three groups: Control, Group A (bleached), and Group B (bleached + artificial saliva). The test showed a statistically significant difference between the groups (p = 0.000).A high F-value of 26.563 indicates that the variation in microhardness is due to the treatment rather than random chance. Bleaching with 16% carbamide peroxide significantly reduced the microhardness of the composite resin, and the differences among the groups were statistically significant.

The Bonferroni post hoc test **(Table 7)** was performed after a significant ANOVA result to determine which group comparisons showed significant differences in microhardness. Both Group A and Group B had significantly lower microhardness compared to the control, confirming that bleaching with 16% carbamide peroxide reduced the surface hardness of the composite. There was no significant difference between Group A and Group B, indicating that post-bleaching storage in artificial saliva did not improve or reverse the reduction in microhardness. The post hoc test confirms that bleaching significantly compromises the microhardness of nanofilled composite resin, and artificial saliva has no restorative effect on this property.

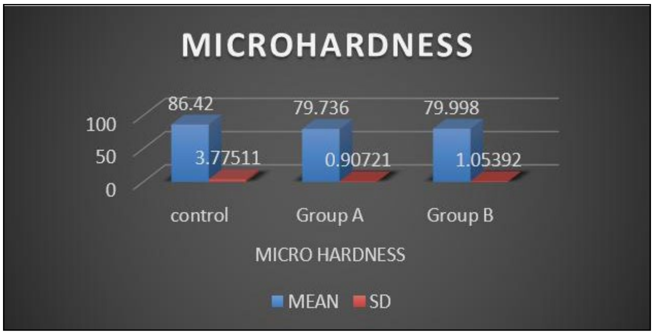
The **(Graph 1)** reinforces the study's conclusion that bleaching with 16% carbamide peroxide weakens the compressive strength of nanofilled composite resin, and post-treatment with artificial saliva does not restore strength.



Graph 1: Compressive strength

The **(Graph 2)** supports the conclusion that 16% carbamide peroxide bleaching reduces the microhardness of nanofilled composite resin.

Artificial saliva immersion does not significantly reverse this effect. The highest hardness was retained by the unbleached control group.



Graph 2: Microhardness

4. Discussion

Cosmetic tooth whitening has significantly evolved since the introduction of nightguard bleaching by Haywood and Heymann in 1989, which utilized carbamide peroxide as the bleaching agent. This technique became popular due to its ease of application, minimal invasiveness, and cost-effectiveness.[4] Since then, various peroxide-based bleaching methods have been employed in clinical practice, primarily using hydrogen peroxide (HP) or carbamide peroxide (CP) delivered through trays, direct application, or in-office procedures. All methods have been proven effective in producing visible tooth whitening. The final level of whitening achieved depends on several factors, including the type of peroxide used, its concentration, and the duration of contact between the active gel and the tooth surface.

While the efficacy of bleaching in improving tooth shade is well-established, emerging evidence has drawn attention to its potential deleterious effects on restorative materials, particularly resin composites. In this context, the present study aimed to evaluate the effect of 16% carbamide peroxide bleaching on the microhardness and compressive strength of nanofilled composite resin (Tetric N-Ceram). The findings demonstrated a significant reduction in both mechanical properties, suggesting that bleaching may compromise the longevity and durability of composite restorations.[4]

The reduction in microhardness observed in the current study aligns with the findings of Kalaivani et al., who reported that exposure of hybrid and microfilled composites to 10% CP for 14 days significantly decreased surface hardness.[5] Similarly, Chakraborty et al. found that both at-home and in-office bleaching agents affected surface microhardness and roughness of resin composites, with the extent of damage depending on the type and filler content of the composite.[6]

Carbamide peroxide consists of two components: the carbamide portion and the hydrogen peroxide portion. When exposed to water, such as saliva in the oral cavity, it breaks down into free carbamide and free hydrogen peroxide. This breakdown is a necessary first step for the bleaching process to occur, as only the released hydrogen peroxide is responsible for the actual whitening effect. Chemically represented as H_2O_2, hydrogen peroxide is an inherently unstable compound that rapidly decomposes into powerful oxidizing agents. These include three types of free radicals hydroxyl radicals, per hydroxyl radicals, and superoxide anions—as well as reactive oxygen species and hydrogen peroxide anions.

1) $CH2NCONH2\ .H2O2 \longrightarrow \text{in water} \longrightarrow H_2NCONH_2 + 2H_2O_2$

Carbamide peroxide — Free carbamide — Hydrogen peroxide

2a) $H_2O_2 \longrightarrow 2HO\bullet$

2b) $HO + H_2O_2 \longrightarrow H_2O + HO\bullet_2$

2c) $HO\bullet_2 \longrightarrow H^+ + O^-\bullet_2$

3) $2H_2O_2 \longrightarrow 2H_2O + 2\{O\} \longrightarrow 2H_2O + O_2$

4) $H_2O_2 \longrightarrow H^+ + HOO^-$

In all of these free radicals, it is the highly reactive and unstable oxygen component that interacts with and oxidizes the long-chain, dark-colored chromophore molecules—structures within a substance that are responsible for its color—found in the enamel of the patient's teeth. This oxidative reaction breaks the chromophores down into smaller, lighter-colored, and more soluble molecules, leading to the visible whitening effect.

Oxidative breakdown of the resin matrix. Hydrogen peroxide, generated from carbomide peroxide decomposition, produces reactive oxygen species (ROS) such as hydroxyl radicals (•OH), perhydroxyl radicals (•OOH), and superoxide anions ($O_2\bullet-$). These highly reactive species attack the carbon–carbon double bonds in the resin matrix, leading to polymer chain scission, as noted by Watts and Addy (2001).[7] This molecular degradation translates clinically into softened surfaces and reduced structural integrity, consistent with the microhardness reductions found in our study.

Lai et al. (2001) reported a significant reduction in shear bond strength of composite resins to enamel immediately after bleaching with 35% HP, attributing it to the presence of residual oxygen which inhibits the polymerization of adhesive monomers.[8] Our study findings are in line with this hypothesis, as the weakening of the resin-filler network and impaired polymerization can also account for the reduced compressive strength observed post-bleaching.

The reduction in microhardness and compressive strength observed in our study correlates with the mechanisms proposed in literature:

1. **Oxidative degradation of the resin matrix:** Free radicals such as hydroxyl and perhydroxyl radicals—released during peroxide decomposition—initiate chain scission of the polymer matrix, leading to a softer and structurally compromised material. This oxidative stress weakens the integrity of the resin structure, consistent with the microhardness reduction noted in our results.
2. **Compromised resin–filler interface:** Our findings align with Lai et al. (2001), who reported that the residual oxygen after bleaching inhibits free radical polymerization, disrupting the bond between resin and filler particles. This ultimately causes microfractures and contributes to a decline in compressive strength, as seen in our results.
3. **Hydrolytic degradation and increased water sorption:** The increased permeability and water absorption following bleaching result in hydrolysis of the resin matrix and resin–filler bonds. These changes are consistent with the observed reduction in compressive strength in this study, especially considering the testing temperature (37°C), which mimics intraoral condition.
4. **Surface porosities and microcrack formation:** Surface irregularities caused by oxidative processes act as initiation sites for stress fractures, thereby compromising both hardness and load-bearing capacity. Surface degradation can propagate through the bulk of the material, explaining the depth-related structural compromise also described in recent bleaching studies.

Furthermore, Francis G. demonstrated that composites exposed to 10% CP for 8 hours daily over 21 days exhibited significant surface deterioration and reduction in flexural strength, which supports our observation that even moderate concentrations and daily exposures can cause clinically relevant material degradation.[9]

The nanofilled composite used in this study, Tetric N-Ceram, contains nano-sized fillers that are designed to enhance polishability, aesthetics, and wear resistance. However, studies by Taher suggest that nanofilled and microhybrid composites are particularly susceptible to bleaching-related damage, likely due to the increased surface area of filler particles and greater resin-filler interface interactions.[10]

While some researchers, such as Turker and Biskin, suggested that bleaching effects may be restricted to the outer surface, others also indicated possible subsurface structural compromise extending up to 2 mm deep, suggesting that simple surface polishing post-bleaching is insufficient to restore original material properties.[11]

Our findings reinforce the depth-dependent degradation concept, indicating that the mechanical deterioration is not superficial and may compromise the long-term load-bearing capacity of restorations.

Interestingly, storage in artificial saliva during the bleaching procedure did not mitigate the adverse effects in our study. The chemical aggression of free radicals on the resin matrix appears to supersede the buffering or remineralizing potential of artificial saliva, at least in the context of restorative materials.[12]

The degree of bleaching-induced damage is closely linked to both concentration and exposure duration. High concentrations and prolonged application can exacerbate material degradation. However, even moderate concentrations, as used in this study, were sufficient to produce clinically significant reductions in both microhardness and compressive strength.

4.1. Clinical implications

1. Restorative procedures should ideally be delayed by at least 1–2 weeks post-bleaching to allow dissipation of residual oxygen and recovery of the enamel surface. This protocol is supported by Titley et al. and Barghi et al. who emphasized that immediate bonding to bleached enamel may result in reduced bond strength and increased restoration failure.[13,14]
2. The application of antioxidants such as sodium ascorbate or glutathione following bleaching has been proposed by Torres et al. to neutralize residual oxygen and restore bonding efficacy, and could be explored in future clinical protocols.[15]
3. In patients with existing composite restorations, bleaching procedures should be approached with caution as shade mismatch, surface roughness, and mechanical weakening may necessitate restoration replacement post-whitening.[16,17]

4.2. Limitations of the study

1. **In vitro nature of the study:** The study was conducted under laboratory conditions, which may not completely replicate the complex oral environment (e.g., temperature fluctuations, saliva flow, masticatory forces, pH changes, bacterial presence).
2. **Short-term evaluation:** Long-term effects, including aging, wear resistance, and repeated bleaching cycles, were not evaluated.
3. **Limited sample size:** Each group consisted of only 10 samples, which, while statistically acceptable, may limit the generalizability of the findings.
4. **Single material and bleaching agent:** Only one type of nanofilled composite resin (Tetric N-Ceram) and a single bleaching agent concentration (16% carbamide peroxide) were tested. The findings may not apply to other restorative materials or different bleaching protocols.
5. **Surface vs subsurface effects:** The study evaluated only surface microhardness and compressive strength. Subsurface changes or alterations in bonding strength to tooth structure were not examined.
6. **No evaluation of other clinical factors:** The study did not assess esthetic changes, color stability, or bond strength to enamel/dentin, which are also clinically relevant when bleaching is done in patients with composite restorations.

5. Conclusions

Within the limitations of this in vitro study, the following conclusions were drawn:

1. A 16% carbamide peroxide bleaching agent had an adverse effect on the micro hardness & compressive strength of Nanofilled composite resin materials
2. The micro hardness reduction in Nanofilled composite resin materials after bleaching was not inhibited by the use of artificial saliva storage media during and after the bleaching procedure.

6. Source of Funding

None.

7. Conflict of Interest

None.

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