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The Effect of a 10% Carbamide Peroxide Bleaching Agent on the Microhardness of Four Types of Direct Resin-based Restorative Materials

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Clinical Relevance

Ten percent carbamide peroxide bleaching agents may minimally reduce the microhardness of microhybrid type of resin-based composite materials compared with a significant microhardness reduction of three other types: nanofilled, silorane-based low shrink, and hybrid.

SUMMARY

Purpose: **This *in vitro* study was undertaken to evaluate the effect of a 10% carbamide peroxide bleaching agent on the microhardness of four types of direct resin-based restorative materials.**

Materials and Methods: **Thirty disk-shaped specimens (10.0 mm diameter × 2.0 mm depth) of each material, including a microhybrid resin composite (Z250), a nanofilled resin composite (Z350), a silorane-based low-shrink resin composite (P90), and a hybrid resin composite (Valux Plus), were fabricated and then polished with medium, fine, and superfine polishing discs. After being polished, specimens were cleaned with distilled water for 2 min in an**

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ultrasonic bath to remove any surface debris and then stored in distilled water at 37°C for 24 hours. Specimens from each material were divided into three groups (n=10). One group was selected as a control group (nontreated 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 top surfaces of the specimens in the different groups were also subjected to the Vickers hardness test with a load of 300 g and 15-second dwell time. Data were analyzed with a one-way analysis of variance and Tukey's HSD test ($\alpha = 0.05$).

Results: **There was a general reduction of Vickers hardness numbers (VHN) values of treated groups compared with the control group for each material used, but this reduction was minimal, with no significant difference between groups in Z250, whereas the other three materials (Z350, P90, and Valux Plus) showed a significant reduction of VHN of treated groups compared with the control group. Conversely, the findings showed no significant difference between treated groups A and B in all materials used except P90.**

Conclusion: **A 10% carbamide peroxide bleaching agent had an adverse effect on the microhardness of nanofilled, silorane-based low-shrink, and hybrid types of resin-based composite materials compared with the microhybrid type.**

INTRODUCTION

Several esthetic procedures have been described in the literature to alter the appearance of smiles, including alterations in the form, texture, position, and color of teeth. The most conservative and noninvasive of these is vital bleaching.[1-3]

All tooth-whitening procedures use either hydrogen peroxide or carbamide peroxide. Currently available home-bleaching agents often contain up to 10% hydrogen peroxide or 22% carbamide peroxide as active ingredients, applied to the teeth via a ready-made or custom-fabricated tray.[4] The most common at-home bleaching agent is 10% carbamide peroxide because of its favorable clinical results, effectiveness, and safety.[3-5]

Patients seeking bleaching agents may have metal- or resin-based or other kinds of restorations in anterior and/or posterior teeth. It is possible that the clinical durability of tooth-colored restorations might be affected by the chemical processes of bleaching agents.[6,7]

The effects of bleaching agents include alterations in the surface morphology and chemical and physical properties of dental restorative materials.[8] One of the most important physical properties of a restorative material is its surface hardness.[9] Surface microhardness is defined as resistance of a material to indentation or penetration.

Several studies that evaluated the effects of bleaching agents on the microhardness of resin-based restorative materials have reported conflicting results. Some found decreased surface microhardness after the bleaching process[10-12] and others did not.[7,13,14]

The conflicting results of the data published about the effects of dental bleaching agents on the microhardness of resin-based composite materials and the resin-based composite restorative materials newly introduced to the market are further reasons for more research to be conducted.

Therefore, this *in vitro* study was undertaken to evaluate the effect of a 10% carbamide peroxide bleaching agent on the microhardness of four types of direct resin-based restorative materials.

The hypothesis tested was that there would be no effect of a 10% carbamide peroxide bleaching agent on the microhardness of four types of direct resin-based restorative materials.

MATERIALS AND METHODS

Specimen Fabrication

The resin-based composite materials evaluated in the current study and their compositions, with the home bleaching agent, are shown in Table 1: (microhybrid) Filtek Z250 (3M ESPE, St Paul, MN, USA), (nanofilled) Filtek Z350 (3M ESPE), (silorane-based low-shrink) Filtek P90 (3M ESPE), and (hybrid) Valux Plus (3M ESPE). For each resin-based composite, 30 disk-shaped specimens (10.0 mm diameter × 2.0 mm depth) were fabricated in shade A3. Cylindrical rubber molds were positioned on a transparent plastic matrix strip lying on a glass plate. The resin-based composite materials were placed in 2.0-mm increments. After the materials were inserted into the mold, a transparent plastic matrix strip was put over them, and a glass plate was secured to flatten the surface. Every specimen was light polymerized for 40 seconds, by means of a halogen light (Elipar 2500, 3M ESPE) within the

Table 1: *Materials Used in This Study*

Material	Composition	Material	Manufacturer
Filtek Z250	Matrix: Bis-GMA, UDMA, and Bis-EMA	Micro-hybrid resin composite	3M ESPE Dental Products, St Paul, MN, USA
	Filler: zirconia/silica (0.01–3.5 μm)		
	Filler by volume: 60%		
Filtek Z350	Matrix: Bis-GMA, UDMA, TEGDMA, and Bis-EMA	Nanofilled resin composite	3M ESPE Dental Products, St Paul, MN, USA
	Filler: Combination of aggregated zirconia/silica cluster filler (0.6-1.4 μm) and nonaggregated 20-nm silica filler		
	Filler volume: 59.5%		
Filtek P90	Matrix: New ring-opening silorane	Low-shrink resin composite	3M ESPE Dental Products, Seefeld, Germany
	Filler: Epoxy functional silane-treated SiO_2 and ytterbium fluoride (0.1–2 μm)		
	Filler volume: 55%		
Valux Plus	Matrix: Bis-GMA and TEGDMA	Hybrid resin composite	3M ESPE Dental Products, St Paul, MN, USA
	Filler: Single filler 100% zirconia/silica (0.01–3.5 μm)		
	Filler volume: 66%		
Opalescence PF	10% carbamide peroxide, potassium nitrate, carbopol, glycerine, 0.11% fluoride ion, flavoring; pH = 6.7	At-home Bleaching	Ultradent Products Inc, South Jordan, UT, USA

Abbreviations: Bis-EMA, bisphenol-A polyethylene glycol dietherdimethacrylate; Bis-GMA, bisphenol-glycidylmethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

range of 480 to 520 mW/cm^2 and verified with a curing radiometer (Optilux Model 100, SDS Kerr, Danbury, CT, USA). The specimens were polished with medium, fine, and superfine polishing discs (Sof-Lex, 3M ESPE) on a slow-speed hand piece rotating in one direction. The final thickness of the polished specimens was verified by a micrometer (Ultra-Cal Mark III, Fowler Tools and Instruments, Sylvac, Newton, MA, USA). After being polished, specimens were subjected to ultrasonic cleaning with distilled water for 2 minutes to remove any surface debris. All specimens were stored in distilled water at 37°C for 24 hours. Specimens from each material were divided randomly into three groups (10/group):

- Control group: Immersed for 14 days in artificial saliva, with no bleaching treatment
- Group A: Treated with a 10% carbamide peroxide bleaching agent for 14 days
- Group B: Treated with a 10% carbamide peroxide bleaching agent for 14 days and then immersed in artificial saliva for 14 days

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 hours at 37°C in preparation for the microhardness test. The artificial saliva was replaced daily**.** Artificial saliva was composed of sodium chloride (NaCl) 0.4 g, potassium chloride (KCl) 0.4 g, calcium chloride ($CaCl_2.H_2O$) 0.795 g, sodium-dihydrogen phosphate ($NaH_2PO_4.H_2O$) 0.69 g, sodium sulfide ($Na_2S.9H_2O$)

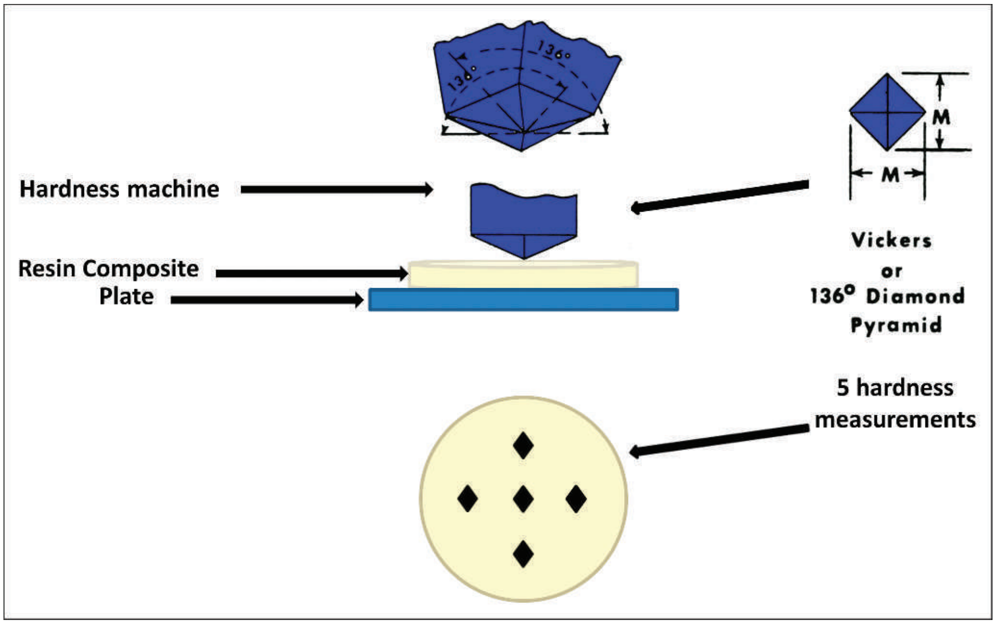

Figure 1. *Schematic showing how specimens were placed under testing facing the Vickers surface hardness diamond indenter.*

0.005 g, and distilled water 1000 mL, and the pH was adjusted to 7.[15]

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 (Opalescence PF 10% Carbamide Peroxide, Ultradent Products Inc, South Jordan, UT, USA). 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 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 test.

Vickers Hardness Test

After the specimens were dried, the Vickers surface hardness test was administered in a universal testing machine (Micromet 2100 series microhardness tester, Buehler, Lake Bluff, IL, USA). 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-second dwell time. Five indentations were made on the top surface and in the middle of each specimen, not closer than 1 mm to the adjacent indentations (Figure 1).

Statistical Analysis

Statistical calculations were performed with SPSS version 16.0 software (SPSS Inc, Chicago, IL, USA). Data were subjected to a one-way analysis of variance (ANOVA) and Tukey's HSD multiple-comparisons test, with the probability for statistical significance set at $\alpha = 0.05$.

RESULTS

Table 2 and Figure 2 show the means and standard deviations of the Vickers Hardness Numbers (VHN) of the specimens in the control group and after the bleaching procedure in the other two groups. The different statistical analyses (one-way ANOVA and Tukey's HSD multiple-comparisons test) are summarized in Table 2.

One-way ANOVA showed a significant difference between the behaviors of the different resin-based composite materials under the different conditions of at-home bleaching in groups A and B compared with the control group ($p<0.0001$).

Table 2: *Vickers Hardness Numbers Means and Standard Deviations of Different Resin-Based Composite Materials Under Different Conditions of Bleaching Compared With Control Group and the Statistical Analysis Summary*[a]

Resin-Based Composite Materials	Resin-Based Composite Types	Vickers Hardness ± SD			One-Way ANOVA	Tukey's HSD Test		
		Group	n	Value	*p* Value	Group	Group	*p* Value
Filtek Z-250	Microhybrid	Control	10	88.22 ± 5.37	<0.0001	Control	A	0.872
		A	10	87.36 ± 2.67		Control	B	0.855
		B	10	87.30 ± 2.92		A	B	0.999
Filtek Z-350	Nanofilled	Control	10	84.64 ± 3.16		Control	A	<0.0001
		A	10	73.98 ± 2.51		Control	B	<0.0001
		B	10	73.74 ± 3.33		A	B	0.983
Filtek P-90	Low-shrink	Control	10	75.68 ± 0.75		Control	A	<0.0001
		A	10	61.76 ± 4.23		Control	B	<0.0001
		B	10	57.46 ± 2.09		A	B	0.005
Valux Plus	Hybrid	Control	10	125.80 ± 5.22		Control	A	<0.0001
		A	10	110.60 ± 4.60		Control	B	<0.0001
		B	10	110.40 ± 3.69		A	B	0.995

[a] *Control = no bleaching; group A = 14-day bleaching; group B = 14-day bleaching followed by 14-day immersion in artificial saliva. Significant difference at* $p \leq 0.05$.

VHN mean values of Filtek Z250 were minimally reduced in the different bleaching groups (A and B) compared with that of the control group, but with no significant difference ($p=0.872$ and $p=0.855$, respectively).

VHN mean values of Filtek Z350 in groups A and B were significantly reduced compared with that of the control group ($p<0.0001$), whereas there was no significant difference between the VHN mean values of groups A and B ($p=0.983$).

VHN mean values of Filtek P90 in the different bleaching groups, A and B, were significantly reduced compared with that of the control group ($p<0.0001$), and the VHN mean value of bleaching group B was significantly reduced compared with that of group A ($p=0.005$).

VHN mean values of Valux Plus in the different bleaching groups, A and B, were significantly reduced compared with that of the control group ($p<0.0001$), whereas there was no significant difference between the VHN mean values of groups A and B ($p=0.995$).

DISCUSSION

Hardness of a material is defined as its resistance to permanent surface indentation or penetration, and this property is related to a material's strength, ductility, elastic stiffness, plasticity, strain, toughness, viscoelasticity, and viscosity. The ability of a material to abrade or to be abraded by opposing dental structures, materials, or any chemical softening has implications for the clinical durability of dental restorations.[16]

The temperature in our study was fixed at 37°C for both bleached and unbleached groups. This was in accordance with the findings of Yu and others[17] who reported that bleaching at 37°C produced a reduction

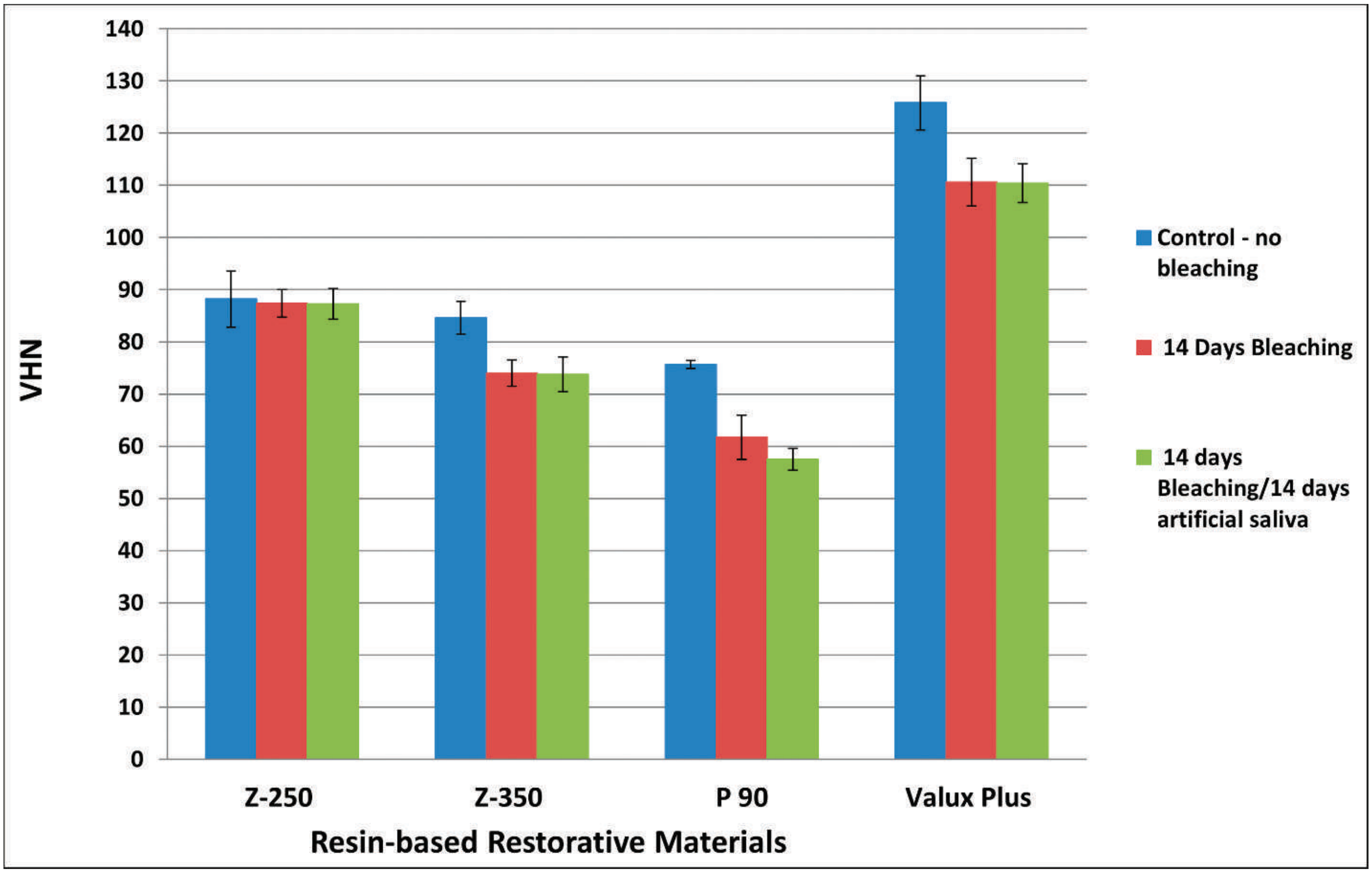

Figure 2. *Vickers hardness numbers means and standard deviations of tested resin-based composite materials under different conditions of bleaching compared with the control group.*

in surface microhardness of composite resin, compomer, and glass-ionomer cement compared with bleaching at 25°C, and that no significant differences were found in the microhardnesses of unbleached groups stored at 25°C and 37°C.

Based on the results of this study, the null hypothesis was rejected, because the use of a 10% carbamide peroxide bleaching agent at 37°C did affect the microhardness of the resin-based restorative materials. This finding is in accordance with those of previous studies, which reported that the microhardness of all dental restorative materials except ceramic was reduced after the application of bleaching agents.[17-19] Conversely, the finding is contrary to those of other studies reporting that the use of bleaching agents had no effect on the microhardness of composite resin, compomer, and glass-ionomer cement.[6,20]

The bleaching gel used in this study contained 10% carbamide peroxide. This type of gel is unstable and degrades immediately into approximately one-third hydrogen peroxide and two-thirds urea on contact with tissue and saliva. After the degradation of carbamide peroxide, the hydrogen peroxide then breaks down into free radicals, which may induce oxidative cleavage of polymer chains and then lead to chemical softening of the dental materials.[6,21] Carbamide peroxide had a greater effect on the microhardness of resin-based composite and glass-ionomer materials at a higher environmental temperature (37°C) compared with a low temperature (25°C), and this might be due to the release of more free radicals at the higher temperature.[17]

The composition of the artificial saliva used in this study acted as an accelerator in the degradation of carbamide peroxide, to mimic the oral environment, and may minimize the adverse effects of bleaching and inhibit microhardness reduction by means of salivary remineralization potential due to the presence of calcium and phosphate ions.[22] In contrast, the findings of this study showed that the use of artificial saliva as a storage medium during and after the bleaching procedure had no benefit, and the reduction in microhardness of the different resin-based composite materials was not inhibited.

The microhardness reduction percentages of the different resin-based composite materials (Filtek Z250, Filtek Z350, Filtek P90, and Valux Plus), after exposure to the same bleaching regimen, were 0.97%, 12.59%, 18.39%, and 12.08%, respectively, which might be related to the different resin matrices, filler contents, and particle sizes. This is in agreement with the results of a study by Hannig and others.[23]

In our study, nanofilled (Z350), silorane-based low-shrink (P90), and hybrid (Valux) resin-based composite materials showed significant reductions in microhardness values after the bleaching procedure, compared with the minimal and nonsignificant reduction in microhardness demonstrated by the microhybrid (Z250) composite.

This might be related to the presence of a higher amount of TEGDMA in the Z350 and Valux Plus resin-based composite materials and the absence of TEGDMA in Z250. The incorporation of TEGDMA diluent monomers in the resin matrix may make the resin matrix less resistant to bleaching agents and may increase the softening of resin composite material.[24] In contrast, the reduction in microhardness of the Z350 was higher than that of Valux Plus, which might be related to the presence of the high molecular weight of the resin matrix and the lower filler content in Z350 compared with Valux Plus.[25]

According to the results, the silorane-based low-shrink (P90) material showed more reduction in microhardness compared with the other tested materials; however, this might be due to the presence of a new ring-opening silorane resin matrix and lower filler content. This type of resin matrix might be softer than other resin matrices (Bis-GMA, UDMA, and Bis-EMA) and easily soluble by bleaching agents.

Furthermore and based on the findings of this current study, bleaching agents should not be used indiscriminately in the patient's mouth, and the teeth that have extensive tooth-colored restorations should not be exposed to bleaching agents or at least protected. Finally, patients should be informed that the physical properties of tooth-colored restorations might be affected by the bleaching procedure, and the restorations might be softened. This could potentially predispose to increased adherence of cariogenic bacteria, surface wear rate, stain absorption, and potential marginal/edge strengths of these restorations and that they may need to be replaced.

CONCLUSIONS

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

1. A 10% carbamide peroxide bleaching agent had an adverse effect on the microhardness of nanofilled, silorane-based low-shrink, and hybrid types of resin-based composite materials compared with the microhybrid type.
2. The microhardness reduction in different resin-based composite materials after bleaching was not inhibited by the use of artificial saliva storage media during and after the bleaching procedure.

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Conflict of Interest

The author of this article certifies that there is no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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