

Effect of 38% carbamide peroxide on the microleakage of silorane-based versus methacrylate-based composite restorations

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Objectives: This study aimed to assess the effect of 38% carbamide peroxide on the microleakage of class V cavities restored with either a silorane-based composite or two methacrylate-based composites. **Materials and Methods:** A total of 96 class V cavities were prepared on the buccal surface of extracted human teeth with both enamel and dentin margins and were randomly assigned into three groups of Filtek P90 (3M-ESPE) + P90 system adhesive (3M-ESPE)(group A), Filtek Z250 (3M-ESPE) + Adper Prompt L-Pop (3M-ESPE)(group B) and Filtek Z350XT (3M-ESPE) + Adper Prompt L-Pop (group C). Half of the teeth were randomly underwent bleaching (38% carbamide peroxide, Day White, Discus Dental, applying for 15 min, twice a day for 14 day) while the remaining half (control) were not bleached. Dye penetration was measured following immersion in basic fuchsin. Data were statistically analyzed using Kruskal-Wallis and Mann-Whitney U tests at a level of 0.05. **Results:** No significant differences were found between composites in the control groups in enamel ($p = 0.171$) or dentin ($p = 0.094$) margins. After bleaching, microleakage of Z250 (in enamel [$p = 0.867$] or dentin [$p = 0.590$] margins) and Z350 (in enamel [$p = 0.445$] or dentin [$p = 0.591$] margins) did not change significantly, but the microleakage of P90 significantly increased in both enamel ($p = 0.042$) and dentin ($p = 0.002$) margins. **Conclusions:** No significant differences were noted between the bleached and control subgroups of two methacrylate-based composites in enamel or dentin margins. Microleakage of silorane-based composite significantly increased after bleaching. (*Restor Dent Endod* 2014;39(3):172-179)

Key words: Microleakage; Silorane composite resins; Tooth bleaching

Introduction

Tooth-colored restorations, especially composite resins are now part of contemporary dentistry.^{1,2} However, drawbacks such as polymerization shrinkage have compromised their clinical success. Polymerization shrinkage will cause gap at the tooth-restoration interface and subsequent microleakage, which allows the passage of fluids, bacteria and ions leading to the development of complications such as hypersensitivity, pulpal irritation and marginal discoloration.²⁻⁴ The following approaches may help to reduce the polymerization shrinkage and the resulting stress: deceleration of polymerization rate, replacement of dual-cured composites with self-cured resins, applying a thicker adhesive coat below the composite resin and application of incremental technique.⁵⁻⁸ Changing the resin matrix and production of composite resins with

small polymerization shrinkage such as silorane-based composites is a recently proposed technique to reduce the polymerization shrinkage.² These composites undergo cationic ring-opening polymerization.^{2,4} New monomers are produced by the reaction of oxirane and siloxane molecules and the name 'silorane' is derived from the names of these two molecules.^{2,3} The manufacturer claims that this composite resin has two main advantages: the first is its small polymerization shrinkage due to the oxirane ring opening mechanism and the second is its increased hydrophobicity attributed to the presence of siloxane.⁴ Studies have demonstrated that silorane-based composites have similar or even more favorable mechanical and physical characteristics compared to methacrylate-based composites: polymerization shrinkage less than 1.5%, low water sorption, optimal biocompatibility, suitable color stability and good marginal fit.⁹⁻¹⁵

Bleaching is an effective and relatively safe esthetic treatment.^{16,17} The bleaching agent usually contains peroxide (such as hydrogen peroxide, carbamide peroxide, and sodium perborate) and is usually applied through the office- or home-bleaching techniques.^{16,18,19} Office bleaching is usually carried out with the use of 35 - 38% hydrogen peroxide applied to the tooth surface for 30 - 45 minutes. This process may be activated by light. Home bleaching is usually done with the application of carbamide peroxide delivered in a special tray customized for the patient; which is usually used at night.¹⁶

Some researchers have investigated the effect of bleaching agents on physicochemical characteristics of tooth structure and some others have evaluated the influence of bleaching materials on the properties of methacrylate-based composite resins such as the elution of methacrylate monomers, their surface hardness and roughness, color and microleakage.¹⁷⁻²² The impact of whitening agents on the surface roughness and hardness and enamel-dentin bond strength of silorane-based composite resins has been evaluated in the literature as well.^{16,23} A group of researchers believe that tooth whitening agents are able to penetrate into the tooth structure through the unsealed dentin margin at the tooth-restoration interface and thus, are capable of causing complications like tooth hypersensitivity and microleakage.^{17,24} However, to date, no study has evaluated the effect of bleaching agents on the microleakage of cavities restored with silorane-based composite resins. On the one hand, Filtek Z350 is a nano-filled composite which has been claimed to have a low shrinkage due to its high filler volume.²⁵ Also, Z250 has had acceptable microleakage results in previous studies.^{26,27} Some manufacturers have produced home bleaching agents with higher concentrations of carbamide peroxide and shorter overnight application time that may have different effects compared to 10 - 15% carbamide peroxide. Therefore, the present

study sought to assess the effect of a highly concentrated home bleaching agent on the microleakage of class V cavities restored with silorane-based composite resins in comparison with two methacrylate-based composites (microhybrid and nanofilled). The null hypothesis is that the effect of bleaching with 38% carbamide peroxide on the microleakage of silorane-based and methacrylate-based composite restorations is the same.

Materials and Methods

The name of products used, their composition and their manufacturing companies are summarized in Table 1. Three types of restorative materials were used in this study: a methacrylate-based microhybrid composite resin (Filtek Z250, 3M-ESPE, St. Paul, MN, USA) and Adper Prompt L-Pop bonding (self-etch adhesive, 3M-ESPE), a methacrylate-based nanofilled composite resin (Filtek Z350, 3M-ESPE) and Adper Prompt L-Pop bonding, and a silorane-based composite resin (Filtek P90, 3M-ESPE) and self-etch bonding and primer (P90 System adhesive, 3M-ESPE). Day white home bleaching system (Discus Dental, Culver City, CA, USA) containing 38% carbamide peroxide was used for bleaching.

Specimen preparation

A total of 96 sound premolar teeth that had recently been extracted as part of orthodontic treatment plan and were free from carious lesions, cracks, fracture, or restorations were selected. The teeth were cleaned from blood and tissue appendages and immersed in 0.5% chloramine T solution at 4°C for one week according to ISO 11405 standards. Then the teeth were polished with a rotary bristle brush and pumice paste and kept in distilled water to be used for the experiment within the next 3 months. Standard class V cavities measuring 3 mm (mesiodistal) x 2 mm (occlusogingival) x 1.5 mm (depth) were prepared by a diamond 008 fissure bur (Stoddard manufacturing, Garden City, England) and high speed hand piece with water spray on the buccal surface of all teeth with gingival margin 1 mm below the cemento-enamel junction. The bur was changed after preparation of each 5 samples.

Material application

The teeth were randomly assigned into three groups as follows:

Filtek Z250 + Prompt L-Pop (PLP)

Filtek Z350 + Prompt L-Pop

Filtek P90 + P90 adhesive

For group A, PLP was mixed according to the manufacturer's instructions and the entire surface of the cavity was coated with the bonding agent using a microbrush and scrubbed

Table 1. Materials and their composition used in the study

Type	Material	Content	Manufacture
Day white	38% Carbamide peroxide	Carbamide Peroxide	Discus Dental, Culver City, CA, USA
Filtek Z250	Microhybrid methacrylate-based composite	Matrix: Bis-GMA, Bis-EMA, UDMA, TEGDMA Filler: zirconia, Silica (0.01 - 3.5 mm), 78 wt%, 60 vo%	3M ESPE, St. Paul, MN, USA
Filtek Z350XT Enamel	Nanofilled methacrylate-based composite	Matrix: Bis-GMA, UDMA, Bis-EMA Filler: Silica, zirconia, nanoparticles (20 µm), and nanoagglomerated) 0.4 - 0.6 µm), 78.5 wt%, 55.9 vol%	3M ESPE, St. Paul, MN, USA
Filtek P90	Silorane-based composite (microhybrid)	Matrix: 3,4 Epoxycyclohexyl ethyl cyclopoly-methylsiloxane, bis-3,4 epoxycyclohexyl-ethyl-phenyl-methylsilane Filler: Silanized, quartz, Yttrium fluoride (0.04 - 1.7 mm) 76% weight, 55% volume	3M ESPE, St. Paul, MN, USA
P90 System Adhesive	Two step self-etch	Primer: phosphorylated-methacrylated, vitrebond copolymer Bis-GMA, HEMA, water, ethanol, silane-treated silica filler, camphorquinone stabilizer Bond: hydrophobic dimethacrylate, phosphorylated-methacrylated, TEGDMA, silane-treated silica filler, initiator, stabilizer	3M ESPE, St. Paul, MN, USA
Adper Prompt L-Pop	Self-etch adhesive	phosphate methacrylate, water, ethanol, silanized colloidal silica, photo initiator	3M ESPE, St. Paul, MN, USA

Bis-GMA, Bisphenol A glycidyl dimethacrylate; Bis-EMA, Ethoxylated Bisphenol A dimethacrylate; UDMA, Urethane dimethacrylate; TEGDMA, Triethylene glycol dimethacrylate; HEMA, 2-Hydroxyethyl methacrylate.

for 15 seconds. After gentle air drying for 5 seconds to thin the adhesive coat, the entire surface was coated with the second layer of adhesive. After gentle air drying for 10 seconds, the adhesive layer was cured with the LED light curing unit (Valo, Ultradent Products Inc., South Jordan, UT, USA) with 1,000 mW/cm² intensity. Z250 composite resin was incrementally applied to the cavity obliquely in three layers. The first layer of composite resin was applied to the occlusal margin and occlusal one-third of axial wall, the second layer was applied to the middle one-third of the axial wall and the third layer to the gingival margin and gingival one-third of the axial wall. Each layer was photo cured for 10 seconds.

Group B specimens were prepared according to the same protocol used for the previous group. Group C specimens were prepared according to the manufacturer's instructions as follows: the entire surface of the cavity was coated with primer using a microbrush and scrubbed for 15 seconds, followed by gentle air drying and light curing with an LED light-curing unit with 1,000 mW/cm² intensity for 10 seconds. By using another microbrush, the entire surface of the cavity was evenly soaked with the bonding agent, followed by gentle air drying and light curing for 10 seconds. The cavities were restored with P90 composite resin with the same technique described earlier.

It should be mentioned that the intensity of the light curing unit was checked after curing every 8 specimens by a radiometer. The specimens were polished by using Soflex aluminum oxide polishing discs (Coarse, medium, fine, super fine, 3M-ESPE) and post-cured for another 20 seconds. The teeth were then immersed in distilled water and stored at 37°C for 24 hours.

Thermocycling

Specimens were subjected to thermocycling for 1,000 cycles between 5°C and 55°C (± 2°C), with a 30 seconds dwell time and a 10 seconds transfer time and then stored in 37°C distilled water.

Bleaching process

Each group was randomly divided into two subgroups of 16 each (A1, A2, B1, B2, C1, and C2). A1, B1 and C1 were immersed in distilled water at 37°C as the control groups and did not undergo the bleaching process. The teeth in A2, B2 and C2 groups were prepared for bleaching as follows: the teeth were first dried and separately fixed on a piece of dental wax on a smooth surface. According to the manufacturer's instructions, 38% carbamide peroxide

(Day White, Discus Dental) was applied twice a day for 15 minutes for a duration of 2 weeks. At each cycle, the restoration surface of samples was covered with the whitening gel, placed in a dark environment for 15 minutes. Samples were then rinsed with water and stored in distilled water at 37°C.

Dye penetration

Apex, root and furcation area were sealed by a layer of adhesive wax. All tooth surfaces were sealed with 2 layers of nail polish except for the restored area and 1 mm around it. The specimens were then immersed in 2% basic fuchsin solution and stored in an incubator at 37°C for 24 hours. The teeth were then washed and dried with sterile gauze and air spray. Each tooth was separately mounted in a special mold using clear polyester resin. After that they were longitudinally sectioned by a cutting machine with 0.82 mm sectioning blade thickness

(Mecatome T201A, Presi, Brié-et-Angonnes, France). Gingival and occlusal marginal microleakage was assessed under a stereomicroscope (Nikon, Tokyo, Japan) with x40 magnification and scored as below (Figure 1):

- 0: No dye penetration
- 1: Dye penetration extending up to ½ of the gingival/occlusal wall
- 2: Dye penetration extending to more than ½ of the gingival/occlusal wall
- 3: Dye penetration extending into the axial wall and pulp

Statistical analysis

Kruskal-Wallis test was used to compare the three composites. If it was significant, Mann Whitney U test with boneferroni adjustment was used for comparisons of two. Whitney U test was used for the comparison of bleached and control subgroups of each composite as well. $p < 0.05$ was considered statistically significant.

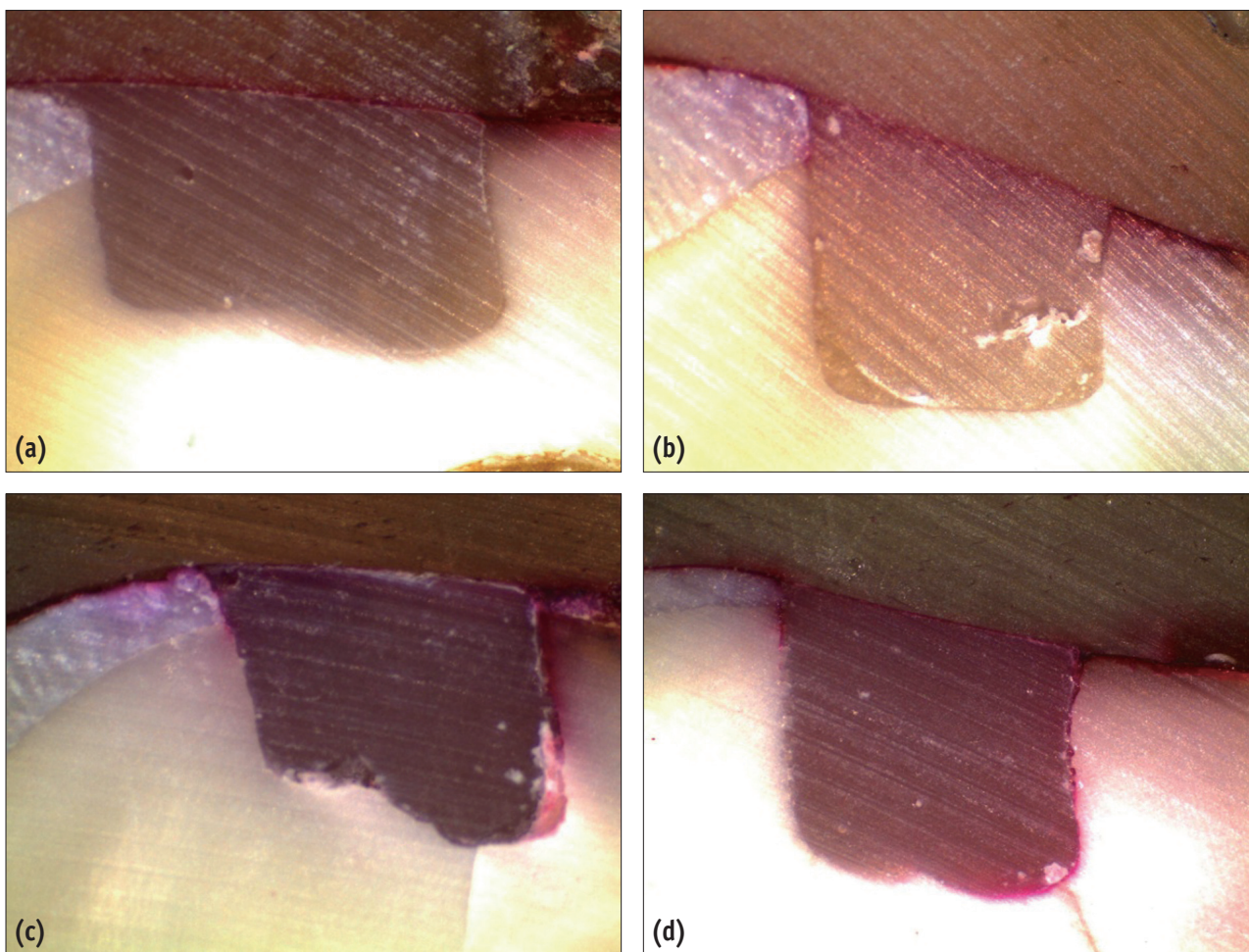


Figure 1. Representative microscopic images showing the scores of microleakage, taken under a stereomicroscope (at a x40 magnification). (a) score 0; (b) score 1; (c) score 2; (d) score 3.

Results

Microleakage scores are given in Table 2. Kruskal-Wallis test indicated no significant differences between the three control groups of Z250, Z350 and P90 composite resins at the enamel ($p = 0.171$) or dentin ($p = 0.094$) margins. After the application of 38% carbamide peroxide, the comparison between the three studied groups in Mann-Whitney U test showed that the microleakage of Z350 was significantly higher than that of Z250 at both enamel ($p = 0.024$) and dentin ($p = 0.027$) margins. P90 also showed significantly higher microleakage than Z250 at both enamel ($p = 0.024$) and dentin ($p < 0.001$) margins. It showed a greater microleakage than Z350 at the dentin margin ($p = 0.014$), but no significant difference was observed at the enamel margin ($p = 0.47$).

Mann-Whitney U test showed no significant differences in the microleakage of Z250 composite in enamel ($p = 0.867$) or dentinal ($p = 0.590$) margins compared to the control group. Also, no significant differences were found in the microleakage of Z350 composite in enamel ($p = 0.445$) or dentinal ($p = 0.591$) margins compared to the control group. However, the microleakage of P90 composite restorations significantly increased in both enamel ($p = 0.042$) and dentin ($p = 0.002$) margins compared to the control group.

No significant differences were noted in the microleakage between the enamel and dentin margins of P90 ($p = 0.863$) and Z250 ($p = 0.058$), but the microleakage of Z350 at enamel margin was significantly greater than dentin margin ($p = 0.011$).

Discussion

Dye penetration is the most commonly used technique for the assessment of the sealability of dental materials under *in vitro* conditions.²⁸ In the oral cavity, dental materials are subjected to thermal changes and the difference in the modulus of thermal conductivity of the tooth and restorative materials causes stress at the interface.²⁹ Thermocycling has been used in the present study to simulate these changes in order to evaluate the effect of bleaching on dental restorations in the mouth. The bond strength of composite to enamel is not as challenging as to dentin.³⁰ Therefore, we have prepared standard class V cavities with occlusal margins located in enamel and gingival margins in dentin, to evaluate the effect of bleaching on microleakage in both enamel and dentin. Bleaching agents are extremely unstable, therefore they produce free radicals that lead to the cleavage of polymer chains and break the double bonds. Additionally, hydrogen peroxide is diffusible.^{16,31}

In the present study, the microleakage of the control group of P90 was not significantly different with the two other control groups in methacrylate-based composites. Similar to our study, Palin *et al.* compared the microleakage and cuspal deflection of MOD cavities filled with EXL596 and Silorane H1 composites, and Filtek Z250 and Z100 methacrylate based composites.³² They found no significant differences between the microleakage of Silorane H1, Filtek Z250 and Z100. Nevertheless, another study done by Hooshmand *et al.* reported lower microleakage of silorane based composites compared to Tetric Evo Ceram and Tetric

Table 2. Number of the samples showing each microleakage score at both occlusal enamel and gingival dentin margins in the control and bleached groups

	Microleakage scores*								<i>p</i> value**	
	Control				bleached					
	0	1	2	3	0	1	2	3		
Occlusal					Occlusal					
Filtek Z250 ^a	8	7	1	0	Filtek Z250 ^c	8	6	1	1	0.867
Filtek Z350 ^a	3	11	1	1	Filtek Z350 ^d	1	12	1	2	0.445
Filtek P90 ^a	4	10	1	0	Filtek P90 ^d	2	7	1	5	0.042
Gingival					Gingival					
Filtek Z250 ^a	11	3	1	1	Filtek Z250 ^e	0	13	1	2	0.590
Filtek Z350 ^a	3	10	0	1	Filtek Z350 ^f	0	5	9	1	0.591
Filtek P90 ^a	8	6	0	1	Filtek P90 ^g	4	1	8	3	0.002

*Same superscript letters in each column show no statistical significant differences between groups. $d > c$, $g > f > e$ ($p < 0.05$ was considered statistically significant).

** The given *p* values are for the comparison of control and bleached groups.

Ceram.³³ There is also another study done by Roula Al-Boni *et al.* which has also shown that class 1 cavities filled with silorane based composites had a lower microleakage compared to Filtek Z250 and Amelogen Plus methacrylate based composites.³⁴ These controversial results in the mentioned studies might have been caused by the kind of material or method used for the tests.

No significant difference was observed in microleakage after application of 38% carbamide peroxide for 15 minutes twice a day between the two methacrylate-based composites and the control groups at both the dentin and enamel margins. Interestingly it was observed that the microleakage of P90 increased after bleaching compared to the control group, but unfortunately there has not been any previous researches available to compare the results with. In a study done by White *et al.* which analyzed the effects of bleaching agents on the microleakage of class 5 cavities filled with Filtek Z250, they found no significant differences among 20% carbamide peroxide, 6% hydrogen peroxide and the control group.³⁵ Nevertheless, Moosavi *et al.*, have compared the effects of 14% carbamide peroxide bleaching agent on class 5 cavities filled by Filtek P60 and RMGI in different time periods and found that the microleakage in P60 dentin margin and RMGI enamel margin significantly increased after bleaching compared to the control group.³⁶

After bleaching with 38% carbamide peroxide, the microleakage in the dentin margin of P90 was greater than that of Z350. The score of microleakage in both margins of P90 was higher than the corresponding value in Z250. A possible explanation for this observation might be related to the properties of their adhesive system. P90 is applied along with a two-step self-etch adhesive commercially named P90 System Adhesive (PSA). First, a self-etching hydrophilic primer (PSA self-etch primer) is applied and separately cured. Then, a hydrophobic adhesive resin (PSA bond) is applied. PSA bond is methacrylate-based and compatible with conventional methacrylate composites.³⁷ This adhesive system has been designed to bridge the gap between the hydrophilic dentin and hydrophobic silorane-based composite. P90 primer and bonding agent are provided in separate bottles and are separately photo-activated, whereas primer and bonding agent of the other two-step self-etch adhesives are mixed on the dentin surface and are photo-cured simultaneously.² A previous study has shown less microleakage in P90 compared to Z250.³⁴ Yamazaki *et al.* has also demonstrated that the microleakage of P90 was less than that of Tetric Ceram after loading but no significant differences were noted without loading.³⁸ In contrast, Palin *et al.* reported that application of silorane-based composite resins in class V restorations did not lead to the development of microleakage in comparison to methacrylate-based composites.³² D'Alpino *et al.* revealed that the self-etch

bonding systems especially that of P90 had a smaller interfacial gap compared to etch and rinse adhesives.² Tezvergil-Mutluay *et al.* has shown that silorane adhesive has had the least microleakage even in association with a methacrylate-based composite which can be due to the high viscosity of the two-layer filled P90 adhesive and it forms a thick adhesive layer on the tooth surface.³⁹ But the bond longevity of P90 is of great concern.⁴⁰ Although, according to the manufacturer's claim, a slight demineralization of tooth structure happens due to the low pH of P90 primer (2.7), we did not see a durable bond after the bleaching of P90.² On the other hand, although we know that PLP is a low-pH self-etch adhesive (0.4), since its primer and adhesive are being applied all at one step, the infiltration of the adhesive is quite equal to the depth of demineralization which has been created by primer.⁴¹ The reason why bleaching in this study did not change the microleakage of restorations can be ascribed to the bond with PLP. It has been claimed and recently been confirmed that P90 primer forms a chemical bond with hydroxyapatite crystals.⁴² It has been documented that optimal bond stability is achievable in silorane-based adhesives. However, there have been concerns regarding the quality and long-term stability of the P90 adhesive hybrid layer.⁴⁰ Santini and Miletic have observed an intermediate zone with 1 micrometer thickness between the primer and the silorane bond with the use of Micro Raman spectroscopy.⁴³ The authors of the mentioned study believed that this area might be the weakest zone of silorane adhesives which causes failure in restorations and further investigations are required in this respect. Considering the claims regarding the presence of chemical bond and one micrometer intermediate zone, it appears that the destructive effects of free radicals released by the carbamide peroxide led to increased microleakage in silorane-based composite resin in the present study. However, we recommend further investigations to evaluate the degradation process of silorane-based composite.

Conclusions

Under the conditions of this *in vitro* study, no significant differences were noted between the bleached and the negative control subgroups of two methacrylate-based composites in enamel or dentin margins. Microleakage of silorane-based composite significantly increased after bleaching.

Conflict of Interest: No potential conflict of interest relevant to this article was reported.

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