### **Dental Research Journal**

#### **Original Article**

# Effect of cyclic loading on microleakage of silorane based composite compared with low shrinkage methacrylate-based composites

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#### ABSTRACT

**Background:** There are many concerns regarding the marginal seal of composite restorations, especially when composite restorations are subjected to cyclic loading. The aim of this study was to evaluate the effect of cyclic loading on the microleakage of silorane based composite compared with low shrinkage methacrylate-based composites in class V cavities.

**Materials and Methods:** In this *in vitro* study, class V cavities were prepared on the facial and lingual surfaces of 48 human premolars (96 cavities). The teeth were randomly divided into four groups of 12 teeth (24 cavities) each and restored as follows: Group I (Siloran System Adhesive + Filtek P90), Group 2 (All Bond SE + Aelite LS Posterior), Group 3 (Futurabond NR + Grandio), and Group 4 (G-Bond + Kalore-GC). All the specimens were thermocycled for 2000 cycles (5–55°C) and then half of the specimens from each group, were Load cycled. All teeth were immersed in 0.5% basic fuchsine dye, sectioned, and observed under a stereomicroscope. Data were analyzed using Wilcoxon test, Kruskal–Wallis, and Mann–Whitney U-tests. P < 0.05 was considered as significant. **Results:** In both unloaded and loaded groups, no statistically significant differences were observed among four composites at the occlusal margin, but a significant difference in gingival microleakage was found between Aelite and silorane. Occlusal and gingival microleakage was not affected by cyclic loading in none of the four restorative materials.

**Conclusion:** Silorane did not provide better marginal seal than the low shrinkage methacrylate-based composites (except Aelite). In addition, cyclic loading did not affect the marginal microleakage of evaluated composite restorations.

Key Words: Composite resins, dental leakage, polymerization, silorane resins

#### INTRODUCTION

Although resin composite materials have improved considerably since their introduction, their polymerization shrinkage remains a problem. This shrinkage could cause tensile stress and consequent debonding at the tooth-composite interface, which



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Website: www.drj.ir www.drjjournal.net www.ncbi.nlm.nih.gov/pmc/journals/1480 may lead to recurrent caries, postoperative sensitivity, and microleakage.<sup>[1]</sup> Several approaches have been proposed to minimize the polymerization shrinkage, such as using an initial low-intensity curing light exposure, incremental placement technique, and

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applying an intermediate low elastic modulus liner.<sup>[1,2]</sup> Use of low-shrinkage composites is one of the other approaches to control polymerization contraction stress.

Silorane, a new class of ring-opening monomers, is derived from the combination of Oxiranes and Siloxanes, combining the properties of both, such as hydrophobicity, biocompatibility and low shrinkage.<sup>[3]</sup> Previous studies have indicated better enamel and dentin marginal integrity of silorane compared to methacrylate-based composites,<sup>[3-5]</sup> while others reported that silorane did not provide better marginal integrity than the methacrylate-based composites.<sup>[6,7]</sup>

Other resin composites (Kalore GC, Grandio and Aelite LS Posterior) used in this study was low shrinkage methacrylate-based composites.

Weakening of the adhesive resin due to cyclic loading is an important issue in restorative dentistry. Some studies reported increased microleakage of the composite restorations under cyclic loading<sup>[8,9]</sup> while others indicated that cyclic loading did not affect the microleakage and marginal integrity of composite restorations.<sup>[10,11]</sup>

Scanning electron microscopic (SEM) evaluation is the gold standard for determination of microleakage in indirect and directly placed adhesive restorations. SEM-investigation on marginal adaptation of class V cavities might be performed easier because of the smaller size of the cavity and is therefore used more commonly.<sup>[12]</sup>

The aim of the current study was to evaluate the effect of cyclic loading on the microleakage of silorane based composite compared with low shrinkage methacrylate-based composites in class V cavities.

#### MATERIALS AND METHODS

In this experimental study 48 extracted intact human maxillary premolars, without caries, cracks or previous restorations were used. The teeth were immersed in 0.5% chloramine T at 4°C for 1 week and then stored in physiologic normal saline solution until use.

Class V cavities (occluso-gingival length of 3 mm, the mesiodistal width of 3 mm, and 1 mm dentinal depth) were prepared on the buccal and lingual surfaces of the teeth using tapered fissure diamond bur (Tizkavan, Tehran, Iran) with water-cooled high-speed handpiece.

A 0.5 mm, 45°C bevel was placed on the enamel margins using a flame-shaped diamond bur (Diatech Dental AG) while gingival margins were prepared at 90°C with the external surface.

A new bur was used for every five preparations. The gingival margins were 1 mm beneath the cementoenamel junction on dentin. The occlusal margins were located on enamel. The prepared teeth were randomly divided into four groups of 12 teeth each (24 cavities).

Materials used in this study with their chemical compositions are listed in Table 1.

In all groups, 37% phosphoric acid gel (Total Etch, Ivoclar Vivadent) was applied to the enamel part of the cavity for 15 s, rinsed for 15 s and excess water was removed with a light air stream to achieve a moist surface and then restored as follows:

- Group 1 (Siloran System Adhesive + Filtek P90): The Silorane Self Ecth Primer (3M ESPE, Dental Product, ST Paul, USA) was applied and agitated on dentinal surfaces of cavity for 15 s, gently air-dried, light-cured for 20 s using a LED light-curing unit (Guilin Woodpecker Medical Instrument Co., China) at 900 mW/cm<sup>2</sup> intensity, as checked with a radiometer (LED Radiometer Demetron, Kerr, USA) after every 10 uses, and the silorane bond was then applied on all surfaces of cavity followed by a gentle stream of air, and cured for 20 s. Then each cavity was filled with Filtek P90 A3.5 shade composite (3M ESPE, Dental Product, ST Paul, USA). In all groups, the cavities were filled in three increments: The first increment on the axial wall, the second increment was placed from about the midpoint of the gingival wall to the occlusal cavosurface margin and the third increment filled the remaining of preparation, and each increment was cured for 40 s
- Group 2 (All Bond SE + Aelite LS Posterior): All Bond SE (Bisco Inc., Schaumburg, USA) was applied and agitated for 10 s, gently air dried for 5 s and then air dried with greater pressure completely. Another layer of bonding was applied and the process was repeated again and light cured for 20 s. Then each cavity was filled with three layers of Aelite LS Posterior A3.5 shade composite (Bisco Inc., Schaumburg, USA) and each increment was cured for 40 s
- Group 3 (Futurabond NR + Grandio): A moderately thin layer of Futurabond NR (Voco

Material	Composition	Manufacturer	Batch number
Silorane System Adhesive	Primer: Phosphorylated methacrylates, Vitrebond copolymer, Bis-GMA, HEMA, water, ethanol, silanetreated silica filler, initiators, stabilizers Bond: Hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators, stabilizers	3M ESPE, Dental Product, ST Paul, USA	20071239
Futurabond NR	Liquid A: Methacryl phosphorus acid ester and carbonic acid modified methacrylic ester Liquid B: Water, ethanol, silicon	Voco Cuxhaven, Germany	610458
All Bond SE	Part I: Ethanol, sodium benzene sulfinate dehydrate Part II: Bis (glyceryl 1,3 dimethyacrylate) phosphate; hydroxyethyl methacrylate, biphenyl dimethacrylate	Bisco Inc, Schaumburg, USA	0600010905
G-Bond	4-MET, phA-m, DMA, ethanol, water, filler, photo-initiator, stabilizer	GC Corporation Tokyo, Japan	0507279
Filtek P90 (Silorane)	Resin matrix: 3,4-eoxycyclohexylethylcyclopolymethylsiloxane, Bis-3,4-poxycyclohexylethylphenylmethyl silane; filler: Silanized quartz; yttrium fluoride; 76 wt%	3M ESPE, Dental Product, ST Paul, USA	195407
Grandio	Resin matrix: Bis-GMA, TEGDMA, filler: Fluorosilicate glass, $SiO_2$	Voco Cuxhaven, Germany	1106467
Aelite LS Posterior	Resin matrix: Ethoxylated bis-GMA Filler: Glass filler, amorphous silica	Bisco Inc, Schaumburg, USA	0900001308
Kalore-GC	Resin matrix: DX-511 monomer, UDMA, dimethacrylate co-monomers Filler: (30-35 wt% prepolymerized filler, 20-30 wt% fluoroaluminosilicate glass, 20-33% wt% strontium/barium glass, 1-5 wt% silicon dioxide nanofiller)	GC Corporation Tokyo, Japan	1004121

#### Table 1: Materials used in this study and their chemical composition

UDMA: Urethane-dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; Bis-GMA: Bisphenol A-glycidyl methacrylate

Cuxhaven, Germany) was applied for 20 s and air dried for 10 s. Another layer of bonding was applied and the process was repeated again and light cured for 20 s. Then each cavity was filled with three layers of Grandio A3.5 shade composite (Voco Cuxhaven, Germany) and each increment was cured for 40 s

 Group 4 (G-Bond + Kalore-GC): G-Bond (GC Corporation, Tokyo, Japan) was applied and left undisturbed for 10 s. Then air dried for 5 s. Another layer of G-bond was applied and the process was repeated again and light cured for 20 s. Then each cavity was filled with three layers of Kalore-GC A3.5 shade composite (GC Corporation, Tokyo, Japan) and each increment was cured for 40 s.

All specimens were finished using fine-grit finishing diamond burs (Diatech Dental AG, Heerbrug, Switzerland) and polished with sequential disks (OptiDisk, Kerr, USA) (15 s in each margin).

After storage in an incubator (Malek-Teb, Iran) at  $37^{\circ}$ C for 24 h, all teeth were subjected to 2000 thermal cycles of  $5^{\circ}$ C/55 $^{\circ}$ C, with a dwell time of 30 s in each bath and a transfer time of 10 s (Malek-Teb, Iran).

Then in each group, half of the teeth (n = 6) were stored in an incubator at 37°C and the others were load cycled (Germany, SD Mekanotronik), as follows: Initially, a cylindrical tube was coated with a layer of wax, then the teeth were mounted up to 1 mm apical to the gingival margin of the restoration in autopolymerized acrylic resin (Acropars, Iran) at the middle and parallel to walls of the tube. Then the specimens were subjected to 200,000 axial cycles of loading at 80 N, a frequency of 2 Hz and a displacement of 1 mm.

## Field emission-scanning electron microscopic replicas preparation

Before sectioning impression the teeth. an from the surface of the restoration (Precise, Coltene, Switzerland) was taken of 32 specimens (4 randomly selected restorations in each subgroup) and positive epoxy resin replica of each specimen (Epo-thin, Buehler Ltd., Lake Bluff, IL, USA) was obtained. Each resin replica was mounted on a metallic stub, sputter-coated with a thin layer of gold and examined under a field emission-SEM (FE-SEM) (Hitachi S-4160, Japan) with ×1000 magnification and interfacial gaps were measured [Figure 1]. The whole length of the gaps was expressed as a percentage of the length of the total restoration margins (enamel and dentin margins).

#### Microleakage evaluation

After SEM replicas preparation, the root apices of the teeth were sealed with sticky wax, and all surfaces of the teeth were covered with two coats of nail



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more microleakage was selected for assessment. The degree of microleakage was scored according to the following criteria:<sup>[13]</sup>

- 0. No dye penetration.
- 1. Dye penetration up to one-half of the occlusal or gingival wall.
- 2. Dye penetration greater than one-half of the occlusal or gingival wall, but not reaching the axial wall.
- 3. Dye penetration along the axial wall.

The Kruskal–Wallis test, Dunn procedure, and Mann–Whitney U-test were used for statistical analysis of the data. The difference between the occlusal and gingival dye penetration scores of each specimen was analyzed by the Wilcoxon test and P < 0.05 was considered statistically significant.

#### RESULTS

Results of occlusal and gingival microleakage are shown in Table 2.

No statistically significant differences were observed between the microleakage of unloaded and loaded groups on both occlusal and gingival margins in all materials (P > 0.05).

In both unloaded and loaded groups, occlusal microleakage among four composites was not significantly different (P = 0.092, P = 1, respectively). However, at the gingival margin, Aelite showed significantly higher microleakage than silorane restorations (P = 0.03, P = 0.005, respectively), and no significant differences were detected between the other groups (P > 0.05).

When comparing the microleakage between occlusal and gingival margins in each group, there were significantly more dye penetration at the gingival margin than the occlusal margin in all the tested groups (P < 0.05), except unloaded and loaded silorane groups (P = 0.19, P = 0.18, respectively).

Table 3 summarizes the interfacial gap formation observed by FE-SEM. Due to the limited sample size (n = 32), the statistical analysis of the data was not performed.

#### DISCUSSION

The main cause for the clinical failure of composite restoration is marginal leakage along the tooth-restoration interface.<sup>[14]</sup>

**Figure 1:** Scanning electron microscopic micrograph of resin-dentin interface of unloaded groups (Silorane (a), Aelite (b), Grandio (c), Kalore-GC (d)) and loaded groups (Silorane (e), Aelite (f), Grandio (g), Kalore-GC (h)).

polish except for 1 mm around the margins of each restoration. All specimens were then immersed in 0.5% basic fuchsine dye for 24 h at 37°C, washed thoroughly with distilled water, air dried and embedded in acrylic resin.

All teeth were then sectioned into two halves longitudinally from buccal to lingual surface through the center of the restored area using a low-speed diamond disk mounted in a cutting machine (Presi, Mecatome, T201A, France) under constant water irrigation.

Dye penetration at the occlusal and gingival margins was blindly assessed in the two halves by two independent investigators using a stereomicroscope (Nikon 800, Tokyo, Japan) at  $\times 10$  and  $\times 40$  magnifications; if the dye penetration score on the two halves was different, the half that showed

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Groups	Occlusal margins (%)				Total (%)		Total (%)			
	Score 0	Score 1	Score 2	Score 3		Score 0	Score 1	Score 2	Score 3	
Grandio										
Unloaded	10 (83)	2 (17)	0 (0)	0 (0)	12 (100)	2 (17)	7 (58)	3 (25)	0 (0)	12 (100)
Loaded	9 (75)	3 (25)	0 (0)	0 (0)	12 (100)	1 (8)	6 (50)	5 (42)	0 (0)	12 (100)
Kalore-GC										
Unloaded	9 (75)	3 (25)	0 (0)	0 (0)	12 (100)	3 (25)	6 (50)	3 (25)	0 (0)	12 (100)
Loaded	9 (75)	3 (25)	0 (0)	0 (0)	12 (100)	1 (8)	7 (58)	4 (34)	0 (0)	12 (100)
Aelite LS Posterior										
Unloaded	9 (75)	3 (25)	0 (0)	0 (0)	12 (100)	2 (17)	5 (42)	4 (34)	1 (8)	12 (100)
Loaded	9 (75)	3 (25)	0 (0)	0 (0)	12 (100)	0 (0)	5 (42)	6 (50)	1 (8)	12 (100)
Silorane										
Unloaded	10 (83)	2 (17)	0 (0)	0 (0)	12 (100)	7 (58)	5 (42)	0 (0)	0 (0)	12 (100)
Loaded	9 (75)	3 (25)	0 (0)	0 (0)	12 (100)	6 (50)	5 (42)	1 (8)	0 (0)	12 (100)
Total	74 (77)	22 (23)	0 (0)	0 (0)	96 (100)	22 (23)	46 (48)	26 (27)	2 (2)	96 (100)

#### Table 2: Microleakage score in each margin according to the tested composite and cyclic loading

#### Table 3: Results of interfacial gap formation observed by field emission scanning electron microscopy

Filling materials	Loading status	The mean percentage of interfacial gaps	Minimum (µm)	Maximum (µm)	SD	95%CI	
		of four specimens of each groups (%)				Lower bound	Upper bound
Grandio	Unloaded	0.42	0	105	0.44	-0.28	1.1
	Loaded	1.29	0	630	2.5	-2.8	5.4
Kalore-GC	Unloaded	0.95	47	177	0.5	0.01	1.9
	Loaded	1.45	37	350	1.08	-0.2	3.1
Aelite LS Posterior	Unloaded	2.91	0	1000	3.7	-2.9	8.7
	Loaded	3.7	0	1200	4.7	-3.8	11.3
Silorane	Unloaded	0.5	16	100	0.3	-0.01	1.01
	Loaded	1.04	0	400	1.5	-1.4	3.5

CI: Confidence interval; SD: Standard deviation

The resin composites used in this study were low-shrinkage composites. Kalore is a nano-hybrid resin composite with the filler content of 82% by weight. This resin composite is based on Dupont technology, which contains a DX511 molecule in its matrix. The DuPont molecule, DX-511, is a urethane dimethacrylate monomer with a low number of C = C double bonds that is compatible with the current bonding systems and composites. The molecular weight of DX-511 is 895 which is twice that of UDMA or Bis-GMA. The low polymerization shrinkage of Kalore (1.7%) is due to the presence of a low number of C = C double bonds and high molecular weight of DX511.<sup>[15,16]</sup>

Aelite LS posterior is a highly filled hybrid resin composite (74% by volume and 88.5% by weight), and its low polymerization shrinkage is due to its high filler content (1.39%).<sup>[17]</sup> Grandio is a highly filled nanohybrid resin composite (71.4% by volume and 87% by weight). Its nanostructure reduces its polymerization shrinkage (1.57%).<sup>[3]</sup>

#### **Margin effect**

In this study, gingival microleakage in Kalore, Grandio and Aelite (in both unloaded and loaded groups) were significantly higher than occlusal margins; this finding was in agreement with the previous studies that indicated less microleakage at the occlusal margin than gingival margin.<sup>[11,13,18]</sup> This was expected as dentin is a less favorable bonding substrate, due to its lower inorganic material (<50%), higher water content (21%), and its tubular structure.<sup>[19]</sup> Moreover, in the current study enamel margins were etched with 37% phosphoric acid before applying self-etch adhesives. Different studies reported better marginal integrity of self-etch adhesives when the adhesive was applied following selective etching of enamel with 37% phosphoric acid.<sup>[20,21]</sup>

There were no significant differences between the occlusal and gingival microleakage in loaded and unloaded silorane groups. This result is probably due to the low-shrinkage nature of silorane and the fact that at the gingival margin, low polymerization shrinkage stress cannot overcome the adhesive strength.<sup>[22]</sup> Silorane primer with almost pH of 2.7 provides a mild etching and slight decalcification of the tooth structure and a strong and long lasting bond.<sup>[23]</sup> Moreover, Mine *et al.* showed that silorane primer forms a chemical bond with hydroxyapatite crystals.<sup>[24]</sup> Furthermore, some studies reported the higher microleakage and lower bond strength of one-step self-etch adhesives.<sup>[25,26]</sup> In the current study, the Silorane system adhesive was two-step self-etch, while the other adhesives were one-step self-etch.

#### **Filling material effect**

In the current study, no statistically significant differences were observed among four groups at the occlusal margin (in both unloaded and loaded groups), which is in accordance with the results of earlier studies.<sup>[18,27]</sup>

There was a statistically significant difference in microleakage between Aelite and silorane at the gingival margin (in both unloaded and loaded groups). This result is in accordance with the result of Boaro *et al.* that reported the higher microleakage of Aelite compared with the other low shrinkage composites (Heliomollor, Venus Diamond, Filtek Z250 and Silorane).<sup>[28]</sup> Calherios *et al.* also reported that the microleakage of class V cavities restored with Aelite LS was higher than that of similar cavities restored with the other low-shrinkage composites.<sup>[29]</sup> According to Hooke's law, polymerization shrinkage and viscoelastic properties of the resin composite.<sup>[17]</sup>

The higher microleakage of Aelite LS is associated with its high elastic modulus and stiffness due to its high filler levels. Its high stiffness offsets its low polymerization shrinkage that results in high-stress values.<sup>[29]</sup> FE-SEM evaluation also confirmed these results by showing higher percentage of interfacial gaps in the specimens restored with Aelite (in both unloaded and loaded groups). However, due to the inadequate sample size (n = 32), the statistical analysis of the data was not performed.

In the present study, there were no significant differences in microleakage between Silorane, Grandio and Kalore; this finding was in agreement with the results of previous studies reporting that Silorane did not provide better marginal integrity than the methacrylate-based composites.<sup>[6,7]</sup> However, Al-Boni *et al.* reported lower microleakage of Class I

cavities restored with Silorane compared with the other composites (Filtek Z250 and Amelogen Plus).<sup>[30]</sup> Joseph et al. also reported that the microleakage of Class II cavities filled with Filtek Silorane was significantly lower than that of similar cavities filled with methacrylate-based composites.[4] These differences in studies may be explained by differences in resin composites and bonding type, microleakage scores, and cavity type. It may also be related to the formation of an oxygen inhibition layer due to the curing of Silorane primer prior to the application of bonding agent. This layer is formed between the cured primer and the Silorane bond and can be observed in micro-Raman spectroscopy as the intermediate zone of approximately 1 µm; which may be the weakest zone of Silorane adhesives (but it is controversial).<sup>[31]</sup>

#### Cyclic loading effect

In the current study, occlusal and gingival microleakage of all the tested materials was not affected by cyclic loading. This finding was in agreement with the results of previous studies,<sup>[10,11,32]</sup> although some studies showed increased microleakage of composite cyclic loading.<sup>[1,8,9]</sup> Campos restorations under et al. evaluated the microleakage of a condensable composite (Surefil) after cyclic loading using 4000 cycles and 150-newton forces and reported that the effect of cyclic loading on gingival and occlusal microleakage was statistically significant.<sup>[8]</sup> Erdilek et al. also evaluated the microleakage of Spectrum TPH and Admira Ormocer after cyclic loading using 50,000 cycles and 50-newton forces and reported that the cyclic loading significantly increased microleakage for both the materials at the gingival margin.<sup>[9]</sup>

It seems that the properties of a resin composite, rather than marginal adhesion are considered as the most influential factor on its resistance to marginal degradation.<sup>[33]</sup> The resin composites used in this study were low-shrinkage composites. It is hypothesized that, since low-shrinkage composites provide lower polymerization stress, they would be able to withstand fatigue at the interface better than the other composites.<sup>[1]</sup>

Another possible explanation is the use of composites with nanofiller content (grandio and kalore are nanohybrid composites). Cyclic loading leads to a decrease in bonding performance due to fatigue at the adhesive interface.<sup>[5]</sup> Some studies reported that nanocomposites with higher compressive strength also had higher fatigue limits.<sup>[34,35]</sup>

Moreover, Grandio and Aelite contain spherical filler particles that have been associated with reduced stress concentration during loading compared with the sharp edges present within irregular-shaped fillers. Irregular-shaped fillers may act as a defect center promoting the accumulation of stress-induced damage.<sup>[36]</sup>

#### CONCLUSION

Under the limitations of the present study, silorane did not provide better marginal seal than the low shrinkage methacrylate-based composites (except Aelite). Cyclic loading did not increase the microleakage of evaluated composite restorations.

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#### **Conflicts of interest**

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article.

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