# MARGINAL ADAPTATION OF A SELF-ETCH ADHESIVE/ SILORANE-BASED RESIN COMPOSITE IN CLASS V RESTORATIONS

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## ABSTRACT

The objective of the study was to evaluate and compare marginal adaptation of a self-etch adhesive/silorane-based resin composite in Class V restorations with that of a total-etch adhesive/ microhybrid methacrylate-based resin composite. Forty freshly extracted premolars were selected for this study. Standardized Class V cavities were prepared on the buccal and lingual aspects of each tooth.The occlusal margin of each cavity was prepared 1 mm above the cemento-enamel junction, while the gingival margin extended 1 mm beyond it onto the root. Prepared teeth were divided randomly into four groups of 10 teeth each. Cavities in Groups 1 and 3 were restored with the total-etch adhesive Adper<sup>™</sup> Single Bond 2/Filtek Z250 microhybrid methacrylate-based composite (controls), whereas self-etchant P90 System Adhesive/Filtek P90 low shrinkage silorane-based composite was used to restore cavities in Groups 2 and 4. After finishing and polishing, teeth in Groups 1 and 2 were thermocycled for 1500 times, while teeth in Groups 3 and 4 were thermocycled for 3000 times. Specimens were then sectioned longitudinally, bucco-lingually through the center of each restoration. Epoxy replicas were made of sectioned surfaces and examined by SEM at x100 to detect marginal gaps  $along \ composite$  / tooth interfaces at occlusal and gingival margins. Two-way Analysis of Variance and Student's t-test analyzed the marginal adaptation data. At the occlusal enamel margins, the mean values of the largest gaps in Groups 2 and 4 were significantly higher than those in Groups 1 and 3 (P = 0.000). By contrast, at the gingival dentin margins, the mean values of the largest gaps were significantly higher in Groups 1 and 3 (P = 0.000). When the numbers of thermal cycles increased from 1500 to 3000, no statistically significant differences were detected in the mean widths of gap formation at both occlusal enamel margins (P = 0.54) and gingival dentin margins (P = 0.19). The results of this study show that none of the restorative materials used is capable of perfectly adapting to the cavity margins.

**Key words:** Marginal adaptation, marginal gap, Class V, low-shrinkage, silorane based, self-etch adhesive, resin composite restorations

#### INTRODUCTION

Resin composites were introduced in the 1960s as an alternative to acrylic resins and silicate cements. As the performance of composite has improved, along with the increasing demand for esthetic perfection, clinicians are encouraged to select resin-based composites. However, it is well-known that these restorative materials are not yet able to guarantee excellent results due to their polymerization shrinkage, which can still be considered the primary negative characteristic.<sup>1,2</sup> Several approaches have been tried to overcome the inherent property of polymerization shrinkage and to produce a so-called "low-shrinkage composite". Expanding monomers based on spiroorthocarbonates (SOC) were first used. However, the SOC formulation reduced mechanical properties as it did not allow for the incorporation with BisGMA resins and polymerization of the resin mixture, which resulted in decreased monomer conversion.<sup>3-5</sup> Epoxy-based monomers were also reported to have significantly reduced shrinkage

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<sup>2</sup> Associate Professor and Chairman, Department of Restorative Dental Sciences, College of Dentistry, King Saud University, PO Box 60169, Riyadh 11545, Kingdom of Saudi Arabia on polymerization compared with methacrylate monomers, but the slow reaction rates of these monomers made them unsuitable for use as a filling material. Comonomer mixtures of SOC and epoxy resins have been considered but, although polymerization shrinkage was reduced due to the combination of low-shrinkage of the epoxy resin and expansion of the ring member SOCs, this combination further increased the curetime of the monomer.<sup>3</sup> Cycloaliphatic epoxy resins (oxiranes) formulated with polyols, such as polytetrahydrofuran, provided resins for dental composites with about one-half of the polymerization shrinkage of Bis-GMA based resins.<sup>6</sup> Despite of the advantages of no air-inhibited surface layer and of high flexural strength, these resins have relatively high water sorption<sup>6-8</sup>, which might contribute to a significant decrease in wear resistance.9

More recently, an oxirane-based resin formulation was proposed to overcome the disadvantages of polymerization shrinkage of resin-based composites.<sup>10</sup> The resin chemistry has been developed from the reaction of oxiranes and siloxane molecules, and termed 'silorane'. Siloxanes are known for their hydrophobicity while the oxirane polymers are known for their low shrinkage and superior stability toward many physical/ chemo-physical forces and influences. The combination of the two molecular building blocks provides a biocompatible, hydrophobic and low shrinking silorane monomer.<sup>4,11</sup> These molecules polymerize by a cationic ring opening mechanism. Some studies have reported a decrease in volumetric shrinkage and a significant improvement of marginal integrity on both enamel and dentin of silorane composite compared to methacrylate-based composites.<sup>4,11</sup>

Current restorative techniques are based on the adhesive properties of the tooth-colored composite materials. The establishment of reliable dentin bonding is one of the major challenges in adhesive dentistry. In spite of significant improvements in adhesive systems, the bonded interface remains the weakest area of tooth-colored composite restorations, resulting in marginal discolorations, poor marginal sealing, microleakage and recurrence of caries, and loss of retention of the restoration that are among frequent clinical consequences.<sup>12,13</sup>

Dental adhesives have continuously evolved over the past 30 years. Classically, dentin adhesives have required three basic steps; demineralization, priming, and application of the adhesive resin to form the hybrid layer zone. Research in this field has focused largely on developing simpler systems that reduce clinical steps and are less sensitive to the application technique.<sup>14</sup>

The aim of this study was to evaluate and compare marginal adaptation of a self-etch adhesive/siloranebased resin composite with that of a total-etch adhesive/microhybrid methacrylate-based resin composite placed in Class V restorations. It was hypothesized that there would be no significant differences in enamel and dentin marginal adaptation between the two composite restorative materials.

#### METHODOLOGY

Forty sound human premolars, extracted for orthodontic treatments, were selected for use in this investigation. Immediately after extraction, all teeth were washed under running water to remove blood and mucus, and scaled to remove calculus and remnants of soft tissues. Teeth were then carefully checked by visual examination for any damage caused during extraction, and were also examined under stereomicroscope (Stereoscopic Zoom Microscope SMZ 100/SMZ 800, Nikon, Kawasaki, Japan) for the presence of any enamel cracks or any structural defects. All defective teeth were discarded. The 40 teeth selected were stored in distilled water with 0.05% thymol at room temperature until ready for use.

Standardized circular Class V cavities (2 mm diameter and 2 mm depth) were prepared on the buccal and lingual aspects of each tooth. A total of 80 cavities were prepared using no. 4 round diamond points (Mid-West Dental Product Corp., Des Plaines, IL, USA) in a watercooled high speed handpiece. The occlusal margin of each cavity was prepared 1 mm above the cementoenamel junction, while the gingival margin extended 1mm beyond it onto the root. Each diamond point was used for the completion of four preparations and then discarded. All enamel and dentin margins were prepared without bevels.

Prepared teeth were divided into two divisions of 20 teeth each according to adhesive system/resin composite combination. In the first division, Filtek Z250 (3M ESPE, St. Paul, MN, USA) microhybrid composite combined with a total-etch adhesive system was used for restoring the cavities. The total-etch adhesive system consisted of Scotchbond<sup>TM</sup> etchant and Adper<sup>TM</sup> Single Bond 2 (3M ESPE). Scotchbond<sup>TM</sup> etchant (35% phosphoric acid) was applied for 15 s and then rinsed for

10 s. Excess water was blotted using a cotton pellet. Adper<sup>™</sup> single bond 2 was applied for 15 s, gently air thinned for 5 s and light cured (Elipar Highlight, 3M ESPE) for 10 s. Filtek Z250 composite was adapted to the cavity walls (single application) and cured for 20 s. In the second division, cavities were restored using Filtek P90 low-shrinkage silorane-based composite combined with P90 System Adhesive (3M ESPE). P90 System Adhesive Self-Etch Primer was applied for 15 s followed by gentle air dispersion and 10 s of light curing. P90 System Adhesive Bond was then applied followed by gentle air dispersion and 10 s of light curing. Filtek P90 composite was adapted to the cavity walls (single application), and cured for 40 s. The adhesive systems and composites were used as recommended by the manufacturer, and were applied to the cavity walls following a standard restorative procedure. The output of the curing light was checked before it was used and then after the completion of every 10 restorations in five teeth.

All restorations were immediately finished and polished with flexible disks (Opti Disc, Kerr-Hawe, Bioggio, Switzerland) under simultaneous water cooling to avoid drying out of the teeth. Restorations were checked with a light microscope (Wild Photomakroskop M400, Heerbrugg, Switzerland) at x20 magnification to ensure that no flashes remain along the margins of the restorations. All restored teeth were stored in distilled water at 37°C for 1 week.

Restored teeth in each division were divided into two groups of 10 each, according to the number of thermal cycles employed. Restored teeth in Groups 1 and 2 were cycled in a thermocycling apparatus (Huber THE 1100/1200, SD Mechatronik GmbH, Feldkirchen-Westerham, Germany) for 1500 times, whereas restored teeth in Groups 3 and 4 were thermocycled for 3000 times. All teeth were cycled between 5°C and 55°C with 30-s dwell and 5-s transit times.

The 40 teeth were each embedded in acrylic resin (Orthoresin, DENTSPLY Int., Woodbridge Ontario, Canada) and sectioned longitudinally in a bucco-lingual direction using a low speed, water-cooled diamond saw (Isomet 2000, BUEHLER Ltd, Lake Bluff, IL, USA) through the center of each restoration creating two halves. Only one of the two halves was used in the study, giving a total of 80 occlusal and 80 gingival restoration margins to be measured for gaps. Impressions of the sectioned surfaces were made using a fast

setting silicone impression material (Aquasil Ultra XLV, DENTSPLY Int.), for the subsequent fabrication of epoxy resin replicas (Devcon 5 minute epoxy, ITW Devcon, Rushden, UK). The replicas were sputter-coated with gold (Polaron E-5200 Energy Beam Sciences, Agawan, MA, USA), and examined by SEM (JSM, 6360LV, JEOL, Tokyo, Japan) at x100 magnification to detect marginal gaps along the composite/tooth interfaces at occlusal and gingival margins. The composite/ tooth interface was divided into three regions and measurements of marginal gap widths in each region were made at four points at x500 magnification. The largest marginal gap width in each region was recorded in micrometers (µm), and the mean gap widths for each of the occlusal and gingival margins were calculated.

Data were analyzed using SPSS pc+ version 16.0 statistical software (SPSS Inc., Chicago, IL, USA). Two-way Analysis of Variance was used to compare the mean values of marginal gaps for occlusal enamel and gingival dentin margins between Groups 1-4. Student's *t*-test for two independent samples was used to compare the mean values of occlusal enamel to gingival dentin marginal gaps within each group. A *p*-value of < 0.05 was considered as statistically significant.

# RESULTS

As illustrated in Figure 1, the mean values of the *largest gaps at occlusal enamel and gingival dentin margins* for Groups 1-4 showed statistically significant differences. At *occlusal enamel margins*, the mean values of the largest gaps in Groups 2 and 4 were significantly greater than those in Groups 1 and 3, whereas no significant differences were found between Groups 2 and 4, and also between Groups 1 and 3 (Tables 1 and 2, Figures 2a-2d). Conversely, the mean values of the largest *gingival dentin gaps* in Groups 1 and 3 were significantly higher than those in Groups 2 and 4. No significant differences were found between Groups 1 and 3, and also between Groups 2 and 4. No significant differences were found between Groups 1 and 3, and also between Groups 2 and 4. Tables 3 and 4, Figures 3a-3d).

When comparing the mean largest gaps at occlusal enamel and gingival dentin margins in each group, the mean largest dentin gaps were found to be significantly higher than those of enamel in Groups 1 and 3. No statistically significant differences were detected in the mean largest gaps at occlusal enamel and gingival dentin margins in Groups 2 and 4 (Table 5).



Fig 1: Mean values of largest gaps for occlusal enamel and gingival dentin margins in Groups 1-4. Group 1: Filtek Z250 at 1500 cycles. Group 2: Filtek P90 at 1500 cycles. Group 3: Filtek Z250 at 3000 cycles. Group 4: Filtek P90 at 3000 cycles.



Fig 2: SEM micrographs that show gaps between composite and enamel margins (original magnifications x500). 2A: Gap of 1.5 µm between composite Z250 and margin in Group 1.2B: Gap of 3.0 µm between composite P90 and margin in Group 2. 2C: Gap of 1.2 µm between composite Z250 and margin in Group 3. 2D: Gap of 3.8 µm between composite P90 and margin in Group 4.



Fig 3: SEM micrographs that show gaps between composite and dentin margins (original magnifications x500). 3A: Gap of 22.7 µm between composite Z250 and margin in Group1. 3B: Gap of 7.2 µm between composite P90 and margin in Group 2. 3C: Gap of 23.5 µm between composite Z250 and margin in Group 3. 3D: Gap of 6.9 µm between composite P90 and margin in Group 4.

#### DISCUSSION

During specimen examination, the SEM technique may introduce errors and artifacts from drying, leading to cracking and distortion. To overcome such problems a replica technique was used, which allows repeated examinations at different intervals as needed.<sup>15,16</sup> Clinical simulation using thermal cycling is also an integral part of marginal adaptation studies, and is usually performed because clinical trials are costly and time consuming. However, the number of cycles used varies in different studies, and thus variation in the number of cycles used in the present study was an additional objective to detect any further change in marginal gap width along the tooth/restoration interface. The current study showed no evident relation between the width of gap formation of composite restorations and the number of cycles being increased from 1500 to 3000. Therefore, it could be assumed that thermal stresses may act rapidly to affect the marginal seal, making prolonged cycling unnecessary.17

The gap formation of resin-based composite restorations is influenced by the adhesive system and the resin-based composite.<sup>16</sup> The results of the present

ENAMEL MARGINS AMONG GROUPS 1-4						
Thermocycles	Groups	Materials	Mean (S.D)	Ν		
1500	1	Filtek Z250	$1.28^{a}(0.42)$	20		
	2	Filtek P90	$2.28^{b}(0.51)$	20		
3000	3	Filtek Z250	$1.38^{a}(0.37)$	20		
	4	Filtek P90	$2.26^{b}(0.40)$	20		

# TABLE1: COMPARISON OF MEAN VALUES (µM) OF THE LARGEST GAPS AT OCCLUSAL

Different superscript letters indicate significant differences

N = number of occlusal enamel margins measured

#### TABLE 2: TWO-WAY ANOVA RESULTS FOR COMPARISONS OF MEAN VALUES (µM) OF THE LARGEST GAPS AT OCCLUSAL ENAMEL MARGINS

Source	Type III Sum of Squares	df	Mean Square	F	<i>p</i> -values
Cycles	0.04	1	0.04	0.246	0.62
Materials	17.69	1	17.69	96.30	$0.000^{*}$
Cycles * Material	0.07	1	0.07	0.385	0.54

\*Significantly different

#### TABLE 3: COMPARISON OF MEAN VALUES (µM) OF THE LARGEST GAPS AT GINGIVAL DENTIN MARGINS AMONG GROUPS 1-4

Thermocycles	Groups	Materials	Mean (S.D)	Ν
1500	1	Filtek Z250	$22.55^{a}(1.19)$	20
	2	Filtek P90	$2.29^{b}(0.63)$	20
3000	3	Filtek Z250	$23.43^{a}(1.04)$	20
	4	Filtek P90	$2.20^{b}(0.59)$	20

Different superscript letters indicate significant differences

N = number of gingival dentin margins measured

# TABLE 4: TWO-WAY ANOVA RESULTS FOR COMPARISONS OF MEAN VALUES $(\mu M)$ OF THE LARGEST GAPS AT GINGIVAL DENTIN MARGINS

Source	Type III Sum of Squares	df	Mean Square	F	<i>p</i> -values
Cycles	3.12	1	3.12	3.86	0.53
Materials	8604.404	1	8604.404	1.06	0.000*
Cycles * Material	4.64	1	4.64	5.73	0.19

\*Significantly different

## TABLE 5: COMPARISON OF AVERAGE VALUES $(\mu M)$ OF "THE MEAN LARGEST GAPS" AT OCCLUSAL ENAMEL AND GINGIVAL DENTIN MARGINS WITHIN GROUPS 1-4

Groups	Cate	gory			
	Enamel mean (S.D)	Dentin mean (S.D)	<i>t</i> -value	<i>p</i> -value	95% confidence intervals for difference of mean
1	1.28(0.42)	22.55(1.19)	-75.42	< 0.0001*	(-21.84, -20.70)
2	2.28(0.51)	2.29(0.63)	-0.07	0.95	(-0.38, 0.35)
3	1.38(0.37)	23.43(1.04)	-89.49	< 0.0001*	(-22.54, -21.54)
4	2.26(0.40)	2.20(0.59)	0.39	0.69	(-0.26, 0.39)
*Cimificant differences within Crowns 1 and 2					

\*Significant difference within Groups 1 and 3

study revealed that the total-etch adhesive system (Adper<sup>™</sup> Single Bond 2)/microhybrid methacrylatebased composite Filtek Z250 had significantly smaller enamel margin gaps when compared to the two-step self-etch adhesive (P90 System Adhesive Bond)/low shrinkage silorane-based composite Filtek P90. Bonding to mineralized enamel with total-etch adhesive systems is reliable, well-accepted and based on stable micromechanical retention from the formation of resin tags that fill enamel microporosities produced by phosphoric acid etching.<sup>18,19</sup> The less desirable enamel margins found in this study for the two-step self-etch adhesive system used with the silorane-based composite Filtek P90, are probably due to the mild acidity of the self-etch monomer (pH = 2.7).<sup>20</sup> Such monomers are not capable of intensely etching the enamel, thus providing a lower degree of demineralization and subsequent resin infiltration when compared to phosphoric acid conditioning.<sup>21</sup> Therefore, prior etching of enamel with phosphoric acid was suggested as it improved the bonding performance of two-step self-etch adhesives.<sup>22</sup> Contrary to the results of the present study, other researchers found that after 6 months enamel gap formation for a self-etch adhesive system was significantly less than for a total-etch adhesive system.<sup>23</sup> This contradiction might be attributed to differences in study methodology used, as the pulp pressure was simulated and the teeth were not subjected to thermal stress.<sup>23</sup>

The results of the present study revealed smaller width values of dentin margin gaps for the two-step selfetch adhesive (P90 System Adhesive Bond)/low shrinkage silorane-based composite Filtek P90 compared to the total-etch adhesive (Adper<sup>TM</sup> Single Bond 2)/ microhybrid methacrylate-based composite Filtek Z250. The two-step self-etch adhesive system of the siloranebased composite is less technically sensitive than the total-etch system. The self-etch primer is also hydrophilic with phosphorylated methacrylates, Bis-GMA, HEMA and water/ethanol as the solvent. The primer prepares the less-mineralized wet dentin/collagen surface for the more hydrophobic bonding resin. This adhesive bond contains hydrophobic bifunctional monomer with the purpose of matching the hydrophobic silorane composite. It may be speculated that this twostep procedure might have an increased bonding quality to dentin when compared with the total-etch technique.24

The total-etch adhesive system is very technique sensitive because it is difficult to achieve and maintain

a proper dentin moisture level, as is supported by several studies.<sup>25,26</sup> Excess water limits the penetration and behavior of adhesive systems, while over drying decreases the wettability of the dentin surface and results in collapse of collagen fibrils. This collapse prevents resin monomers from penetrating into deep areas, creating porosities and submicron matrix spaces, leaving unprotected exposed collagen that degrades over time to increase the risk of adhesive failure.<sup>25,27-30</sup> This technique sensitivity may also explain the larger dentin gap values observed in the present study when compared to the enamel gap values for the total-etch adhesive (Adper<sup>TM</sup> Single Bond 2)/microhybrid methacrylate-based composite Filtek Z250.

The literature has shown contradictory results regarding dentin gap formation. Some studies, in agreement with the results of the present study, showed a better marginal adaptation of self-etch than total-etch adhesive systems.<sup>22,31</sup> By contrast, another study reported a better performance of total-etch than self-etch adhesive systems.<sup>32</sup> These differences could be related to variations in the adhesive systems tested, which originate from different chemical compositions, rather than to the technique sensitivity of the total-etch adhesive systems.

Another factor that may contribute to the smaller gap width values at dentin margins of the siloranebased restorations is the material's chemistry. Because of the free-radical polymerization of methacrylate-based composites, volumetric shrinkage ranges from 2-5% with increased stresses around the tooth/restoration interface that can separate the restoration from the tooth at the weak bonding interface.<sup>24</sup> This polymerization shrinkage is an intrinsic property of the resin matrix. Therefore, a different resin matrix system is desirable to reduce volumetric shrinkage.<sup>33</sup>

The volumetric shrinkage of silorane-based composite was determined to be 0.99% when using the Archimedes method.<sup>34</sup> The system uses photo-ringopening cationic polymerization chemistry instead of free-radical polymerization of dimethacrylate monomer. Compared with the linear-reactive groups of methacrylates, the ring-opening chemistry of the siloranes starts with the opening of the ring systems. This process gains space and counteracts to some extent the loss of volume that occurs when the chemical bonds are produced. Overall, the oxirane ringopening polymerization process yields a reduced volumetric shrinkage.<sup>33</sup> Therefore, significantly lower polymerization shrinkage and lower stress development occur. This also explains the decrease in cuspal deflection and the superior marginal integrity of siloranebased composites to methacrylate-based systems reported in other studies.<sup>35,36</sup> Moreover, it explains the insignificant difference that was found between enamel and dentin gap values with the silorane-based composite Filtek P90. Therefore, since silorane-based technology offers lower polymerization shrinkage and related stresses than methacrylate-based technology, materials based on the former should be able to resist thermal stresses at the restoration/margin interfaces better than the latter.

It is difficult to correlate laboratory findings with the clinical behavior of restorative materials. In natural vital teeth, pulp pressure and intertubular fluid flow have a significant influence on dentin moisture levels, thus significantly affecting the composite/restoration interface. Therefore, the use of thermocycling as a laboratory simulation of clinical circumstances may not reproduce the actual consequences of temperature changes and different functional stresses in various clinical environments.

#### CONCLUSIONS

The null hypothesis that there were no significant differences in enamel and dentin marginal gaps between a two-step self-etch adhesive (P90 System Adhesive)/low-shrinkage silorane-based composite Filtek P90 and a total-etch adhesive (Adper<sup>TM</sup> Single Bond 2)/ microhybrid methacrylate-based composite Filtek Z250 was not accepted. None of the adhesive/restorative materials used was capable of perfectly adapting to the Class V cavity margins. The smallest marginal gap values along the gingival dentin margins was demonstrated with the Silorane-based composite Filtek P90, while the microhybrid methacrylate-based composite Filtek Z250 showed the smallest marginal gap values along the occlusal enamel margins. An increase in thermocycling from 1500 to 3000 cycles did not affect marginal adaptation.

#### Acknowledgments

We would like to express our sincere thanks to Prof Khamis Hasan, Prof Ali El-Sahn and Dr Hend Al-Nahedh for their constant guidance and help. Our appreciation is extended to Dr Sheikh Shaffi for carrying out the statistical analysis and to Mr Nassr Maflehi for his help and assistance.

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