

Original Article

Comparison of microleakage in Class II cavities restored with silorane-based and methacrylate-based composite resins using different restorative techniques over time

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ABSTRACT

Background: Despite the growing tendency toward tooth-colored restorations in dentistry, polymerization shrinkage and subsequent marginal microleakage remains a problem. The aim of this *in vitro* study was to compare microleakage between silorane-based and methacrylate-based composite resins at different time intervals and with different restorative techniques.

Materials and Methods: In this *in vitro* study, 108 sound extracted human molar teeth were used. Mesial and distal proximal class II boxes with dimensions of 1.5 mm depth and 4 mm width were prepared. The gingival margins of all cavities were 1 mm below the cement enamel junction. The teeth were randomly divided into three groups based on test materials. In the first group, the teeth were restored by a nanocomposite (Filtek Z350XT, 3MESPE) and SE Bond adhesive (Kuraray, Japan), in the second group, the teeth were restored with a silorane-based (Filtek P90, 3MESPE) and Filtek P90 Adhesive (3M ESPE, USA) and in the third group, the teeth were restored with a microhybrid posterior composite resin (Filtek P60, 3MESPE) and SE Bond adhesive (Kuraray, Japan). Half of the proximal cavities in each of these three groups were restored in two horizontal layers and the other half in four horizontal layers. After a period of aging (24-h, 3-month and 6-month) in water and then application of 500 thermal cycles, the teeth were immersed for 24-h in 0.5% fuchsin and evaluated under a stereomicroscope at $\times 36$ magnification to evaluate leakage in gingival margin. Data was statistically analyzed using Kruskal-Wallis and Mann-Whitney U-tests. $P \leq 0.05$ was considered as significant.

Results: In Z350XT statistically significant differences were observed in microleakage in comparison of 24-h and 6-month intervals ($P = 0.01$) that was higher in 6-month. Comparison of microleakage in P90 and P60 composite resins was also statistically significant and was less in P90. Microleakage was not significantly different between P90 and Z350XT at 24-h. However, this difference was significant at 3-month and 6-month intervals. Differences in microleakage of P60 and Z-350XT composite resins were not statistically significant in all intervals ($P = 0.38$). P90 showed the lowest microleakage during storage in water. Z350XT had microleakage similar to P90 within 24-h, but after 6-month of storage in water, it showed the highest microleakage among all the groups. The number of layers (2 layers vs. 4 layers) did not result in any differences in microleakage scores of the composite resins ($P = 0.42$).

Conclusion: Water storage times did not result in any significant effect on microleakage of P90 and P60.

Key Words: Methacrylate-based composite, microleakage, silorane-based composite, storage

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INTRODUCTION

In recent decades, there has been a growing tendency toward tooth-colored restorations.^[1] Among the disadvantages associated with the use of composite resins is polymerization shrinkage that leads to

accumulation of stresses within the material and at the restoration-tooth interface and if the stress level is higher than the bond strength, gaps are formed at the interface and microleakage occurs,^[2,3] resulting in problems such as post-operative sensitivity, marginal destruction, color change of restoration, recurrent caries, enamel cracks, pulpal inflammation and cusp flexure.^[4,5]

Polymerization shrinkage stress is associated not only with adhesive systems and restorative materials, but also it depends on the C-factor, material placement technique, resin composition, particle size and many other factors.^[6]

Efforts made to improve the clinical efficiency and eliminate internal stresses formed during polymerization of methacrylate-based composite resins have led to the creation of new monomers such as siloranes and new nanoparticle fillers.^[7] Silorane-based composite resins are formed by reactions between oxirane and siloxane molecules; this type of composite resin has the capability of the polymerization reaction in the form of ring opening; therefore, the polymerization shrinkage is minimal. In addition, presence of siloxane results in a lack of solubility in the oral fluids and hydrophobic properties of the material increases.^[1-4,7-9] While the methacrylate-based composites exhibit 2.3-3% of volumetric shrinkage,^[10] this rate has been reported to be approximately 0.9% for silorane-based composite resin, which results in less stress on the cavity walls.^[11]

Nanocomposites contain nanomeric and nanocluster particles as inorganic filler. Nanomeric particles are 20-75-nm-sized non-aggregated silica particles with uniform distribution. Nanoclusters are aggregations of spherical particles of silica and zirconia, 2-20 nm in size. Clusters have micron-sized porosities that are infiltrated by silane coupling agents, so that chemical bonding to the organic matrix is established.^[7] The nanoclusters offer better reinforcing action compared with the microfill or nanohybrid systems.^[12] Nanocomposites has low polymerization shrinkage.^[13]

C-factor is another feature that has effects on composite resins shrinkage control. In cavities with higher C-factors, the potential for plastic deformation and therefore resulting stress is reduced.

Method of placing composite resin in the cavity is important for developing stresses within the material.^[14] The method used to reduce polymerization shrinkage of composite resins is the placement in small layers, which reduces stresses on

the cavity walls during polymerization and increases curing depth.^[15]

Comparison of different methods of composite resin placement in layers has shown that the bond of the first layer is the most important and only when the first layer is bonded to the floor of the cavity, acceptable bond strength is achieved. Filling the cavities with vertically and diagonally placed layers often results in no adhesion to the cavity floor. This may be due to the fact that in horizontal cavity filling method the plugger is firmly adapted to the cavity floor, whereas in the vertical placement technique it is difficult and creates voids at the interface under stress. In deep cavities with high C-factor, horizontal layering method is the most appropriate way to obtain a sufficient bond with the cavity to floor.^[16]

The aim of this study was to compare microleakage in methacrylate-based and silorane-based composite resins (a nanocomposite and a posterior microhybrid composite) at different time intervals using different techniques to fill the cavities. The null hypothesis tested was that nanocomposite resins and silorane-based composite resins do not differ in microleakage test.

MATERIALS AND METHODS

In this *in vitro* study, a total of 108 extracted human molars were stored in glass containers of 1% chloramine T solution for 24-h, then in distilled water at 4°C before use.

The teeth were cleaned with periodontal curettes to remove residual debris and tissue tags on tooth surfaces to reveal the cement enamel junction (CEJ). The apical foramina of the teeth were sealed with sticky wax to prevent dye leakage during the test. Proximal Class II cavities with dimensions of 1.5 mm depth and 4 mm width were prepared on the mesial and distal surfaces of the teeth with 008 fissure burs (D&Z, Germany) with water spray at high speed. The gingival floor was placed 1 mm below the CEJ. After preparation of each eight cavities, a new bur was used in order to maintain the shearing efficiency. Metal strip and matrix holder was used to insert composite resin. In order to avoid extension of material at the gingival margin; the matrix was tightened and held by finger against the gingival margin of the cavities.

Each group contained six teeth (12 proximal cavities) and managed as follows:

Group I

Primer of “Filtek P90 Adhesive” (3M ESPE, USA) was applied to the cavity by a microbrush and was distributed with gentle air pressure and light-cured with light-emitting diode (LED) (Demi LED Light Curing System, Kerr Corp., Orange, CA, USA) at 1100-1200 mW/cm² light-curing unit for 10 s; then bonding of “Filtek P90 Adhesive” (3M ESPE) was applied and distributed with gentle air pressure and light-cured for 10 s. The first layer of Filtek P90 (Filtek P90, shade A3, 3M ESPE) was placed in the gingival floor, with a thickness of approximately 1 mm and light-cured for 40 s; the three other layers (thickness of each layer was approximately 2 mm) were exposed for 20 s each.

Group II

The procedure was similar to those in Group I except that the Filtek P90 composite resin was applied only in two layers (thickness of each layer was approximately 3/5 mm) and each layer was light-cured for 40 s.

Group III

Initially, SE Bond primer (Kuraray Co., Ltd., Tokyo, Japan) was applied to the cavity by a microbrush and was distributed with gentle air pressure; then SE Bond adhesive was applied and distributed with gentle air pressure and light-cured for 20 s. The first Filtek Z350XT (shade A3, 3M ESPE) nanocomposite layer had a thickness of approximately 1 mm, which was applied in the gingival floor and cured for 40 s; three other layers were exposed for 20 s each. The thickness of layers was measured by a periodontal probe.

Group IV

The procedures were similar to those in Group III except that Filtek Z350XT nanocomposite resin was applied in two layers and each layer was exposed for 40 s.

Group V

The procedures were similar to those in Group III except that Filtek P60 (shade A3, 3M ESPE) microhybrid composite resin was used.

Group VI

The procedures were similar to those in Group IV except that Filtek P60 micro-hybrid composite resin was used.

Composite resins were placed in all the cavities horizontally. The test materials are shown in Table 1. During the aging period (6-month, 3-month and 24-h), the samples were stored in distilled water at 37°C in an incubator (Behdad, Tehran, Iran) and then according to ISO 11450 guidelines were subjected to 500 thermal cycles at a temperature of 5-55°C, lasting 30 s in each bath, with 15 s between the baths.

To prevent dye penetration, two layers of nail varnish were used on all the tooth surfaces, except on a distance of 1 mm around the tooth-composite resin interface.

Then the teeth were immersed in 0.5% basic fuchsin dye for 24-h and were then washed under running water for 5 min and divided into two halves with a diamond disc (D&Z Germany) at low speed.

Table 1: Materials used in this study

| Materials | Composition |
|--------------------------|---|
| Filtek silorane adhesive | |
| Self-etching/primer | HEMA, Bis-GMA, water, ethanol, phosphoric acid — methacryloxy-hexylesters mixture, silane treated silica, 1,6-hexanediol dimethacrylate, copolymer of acrylic and itaconic acid, (dimethylamino) ethyl methacrylate, dl-camphorquinone, phosphine oxide |
| Bond | Substituted dimethacrylate, silane treated silica, TEGDMA, phosphoric acid methacryloxy-hexylesters, dl-camphorquinone, 1,6-hexanediol dimethacrylate |
| Clearfil SE Bond | |
| Self-etching/primer | Hydroxyethylmethacrylate, methacryloyloxydecyl dihydrogenphosphate, hydrophilic dimethacrylate, DL-camphorquinone, N, N-diethanol-p-toluidine, water |
| Bond | Hydroxyethylmethacrylate, bisphenyl glycidylmethacrylate, methacryloyloxydecyl dihydrogen phosphate, hydrophilic dimethacrylate, DL-camphorquinone, N, N-diethanol-p-toluidine, silanated colloidal silica (10%, microthin) |
| Filtek P60 composite | Filler: 61 vol% silica/zirconia filler with mean particle size of 0.01-3.5 µm. Matrix: Bis-GMA, UDMA, Bis-EMA resins |
| Filtek P90 composite | Filler: 55 vol% silanized quartz, yttriumfluoride. Matrix: 3,4-epoxycyclohexyl ethyl cyclo polymethylsiloxane, Bis-3,4-epoxycyclohexyl ethyl phenyl methyl silane |
| Filtek Z350 XT composite | Filler: 63.3 vol%: 5-20 nm nonagglomerated silica, 5-20 nm zirconium/silica nanoagglomerate, 0.6-1.4 mm agglomerated particles Matrix: Bis-GMA, UDMA, TEGDMA, PEGDMA, Bis-EMA |

Bis-GMA: Bisphenol A-glycidyl methacrylate; PEGDMA: Polyethylene glycol dimethacrylate; TEGDMA: Triethyleneglycol dimethacrylate; HEMA: Hydroxyethyl methacrylate, Bis-EMA: Bisphenol A ethoxylated dimethacrylate, UDMA: Urethane dimethacrylate

Then all the sections were observed under a stereomicroscope (MGC-IO, Russia) at $\times 36$ magnification. Deepest dye penetration for each half of the sections was scored. Dye penetration was evaluated according to a 4-point scale:

0 = No dye penetration

1 = Dye penetration to the outer half of the gingival floor

2 = Dye penetration to the inner half of the gingival floor

3 = Dye penetration beyond the floor, reaching the axial wall.

At the end of 6-month storage in water, two restorations were selected from each group and observed by scanning electron microscope (SEM). For SEM examinations, the specimens were fixed in 2% glutaraldehyde, dehydrated in ascending concentrations of ethanol series (50, 60, 70, 80, 90, 96 and 100%), dried in the oven, were sputter-coated with gold and examined with SEM (VEGA\TESCAN-LMU, USA) at magnifications of $\times 1500$ ^[17] [Figure 1].

Data analysis

Data were evaluated using Kruskal-Wallis test ($P \leq 0.05$) at a confidence level of 95% to detect any significant differences between the groups. Further analyses were carried out with Mann-Whitney test for pair-wise comparisons between the groups ($P \leq 0.05$) at 95% confidence level.

RESULTS

The difference in the number of composite layers placed horizontally (2 layers vs. 4 layers) did not result in significant differences in microleakage ($P = 0.42$).

P60 composite resin showed no significant differences between all the storage times (24-h, 3-month and 6-month) ($P > 0.05$). Similar results were also observed with P90 composite resin ($P > 0.05$).

Comparison of microleakage of Z350XT composite resin at 24-h and 3-month and 3-month and 6-month intervals showed no significant differences. However, comparison of 24-h and 6-month intervals revealed significant differences.

Comparison of the microleakage between P60 and P90 at 24-h, 3-month and 6-month showed significant differences except for the groups where these composite resins were placed in two layers at 6-month interval ($P = 0.24$) but during this time the P90 placed in four layers revealed lower microleakage ($P = 0.02$).

In comparison of microleakage between P60 and Z350XT composite resins at 24-h, 3-month and 6-month intervals, the differences were not statistically significant. Microleakage between P90 and Z350XT composite resins at 24-h interval was not significantly different ($P > 0.05$). However, there was a difference at 3-month and 6-month intervals ($P < 0.05$).

The results are shown in Figure 2.

DISCUSSION

All the tested groups showed some dye penetration at tooth-restoration interfaces, which can be attributed

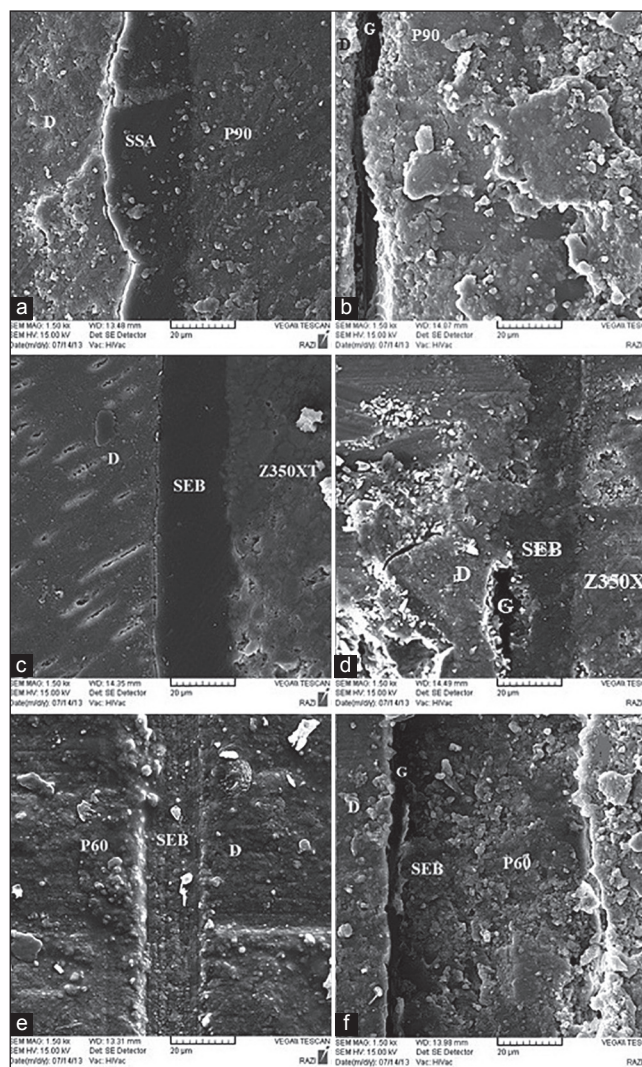


Figure 1: Scanning electron microscope micrographs of microleakage showing tooth-composite resin interface. (a and b) Represent adhesive (Silorane System Adhesive = SSA) and composite resin (Filtek P90). (c and d) Represent adhesive (SE Bond = SEB) and composite resin (Filtek Z350XT). (e and f) Represent adhesive (SE Bond) and composite resin (Filtek P60). In all micrographs D means Dentin.

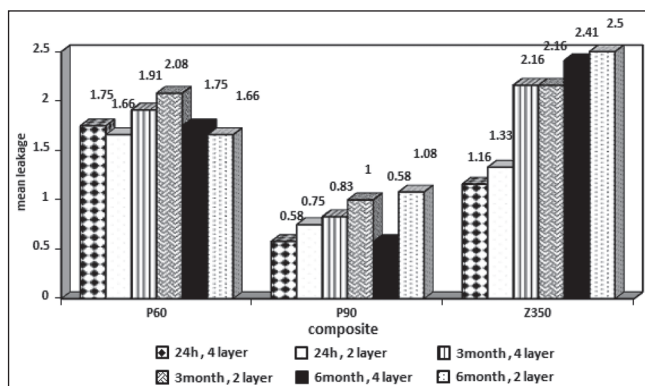


Figure 2: Mean microleakage scores in the different study groups

to dimensional changes resulting from polymerization shrinkage of restorative resins and the differences in thermal expansion coefficients between the teeth and restorative materials. These changes result in internal forces in composite resin materials, causing gap formation at tooth-restoration interface; therefore, microleakage occurs.^[18] Water or ethanol-based three-step etch-and-rinse adhesives are known as standard adhesives in terms of bond durability. Following simplification of application steps of any adhesives, bond durability has decreased and only two-step self-etch adhesives have remained close to standard adhesives.^[19] Self-etch adhesives provide superior and more predictable bond strength to dentin and are, consequently, recommended for direct composite resin restorations, especially when predominantly supported by dentin.^[20] Therefore, in this study, Clearfil SE Bond mild two-step self-etch adhesive was evaluated. This adhesive has shown excellent *in vitro* and *in vivo* performance and therefore can be considered as “standard” among the self-etch adhesives.^[21]

Since in restoring Class II lesions, the use of a metal matrix band along with horizontal insertion of composite materials is a widely used method by dentists and clinical studies, in the present study, this method was used to restore cavities.

In the present study, no difference was observed in microleakage between the cavities restored with various C-factors (different number of layers), similar to the results of studies by Szep *et al.*^[22] Ghavamnasiri *et al.*,^[23] Aranha and Pimenta^[24] and St George *et al.*^[25]

In the present study, although silorane-based composite resin showed the lowest scores of microleakage at 24-h, it was not able to fully prevent microleakage, consistent with the results of a study by Bogra *et al.*^[26]

However, a study by Bagis *et al.* showed that wide Class II cavities restored with silorane composite resin exhibited no microleakage and the margins were completely sealed. This variation could be due to differences in assessment magnifications. In the present study $\times 36$ magnification was used and in Bagis *et al.* study $\times 10$ magnification was used.^[27] In addition, in a study conducted by Usha *et al.* silorane-based composite resin, regardless of the method used to repair Class V cavities (split incremental approach or oblique incremental), showed some microleakage.^[28]

Coefficients of thermal expansion in enamel, dentin and composite resin material are $17 \times 10^{-6}/^{\circ}\text{C}$, $11 \times 10^{-6}/^{\circ}\text{C}$ and a in a range between $20 \times 10^{-6}/^{\circ}\text{C}$ and $80 \times 10^{-6}/^{\circ}\text{C}$, respectively. The difference between the coefficients of thermal expansions and contractions affect the methacrylate-based composite resins more than silorane-based composite resins and it would increase marginal gap formation in methacrylate-based composite resins (Filtek P60 and Filtek Z-350XT) after thermal cycling compared with silorane-based composite resin (Filtek P90).^[29]

There was no significant difference in the degree of microleakage between nanocomposite Z350XT and P60, which is due to approximately similar filler loading (61% and 63% by volume) and similar resin composition and therefore similar elastic modulus and consequently contraction stress. Similar results were reported by Cara *et al.*^[30] and Kusgoz *et al.*^[31] In these studies P60 showed microleakage similar to nanocomposites. Water storage times did not showed any statistically significant effect on microleakage of silorane-based (P90) and microhybrid composite (P60), which was in agreement with study by Mahmoud and Al-Wakeel Eel.^[32]

Silorane-based dental composites showed the best results at the end of the aging period. This is probably due to their unique and low-shrinkage matrix and presence of fillers in the adhesive system. This filler-containing adhesive creates the relatively strong hybrid layer which provides hydrolytic stability in the long term. In silorane adhesive system, the primer and bonding component are separately light-cured; in order to match with the hydrophobic silorane composite resin, the bonding agent has hydrophobic bifunctional monomers in its composition. This is probably the reason why this two-step procedure can improve the quality of tooth-composite resin interfaces. Water absorption in silorane composite resin is less than that

in conventional methacrylate-based composites because hydrophobic siloxane backbone can be effective in reducing the washing and removal of unpolymerized monomer from the resin matrix.^[33] Silorane light polymerization is cationic and has a greater affinity for oxygen compared to free radical polymerization and does not form an air-inhibited layer. Therefore, not only polymerization shrinkage decreases, but also due to this effect, the degree of conversion in silorane adhesive component increases.^[34] Thus, water absorption of the silorane composite resin is low and this also has been reported by Kopperud *et al.*^[35] Porto *et al.*^[36] and Schneider *et al.*^[37]

After 6-month immersing the samples in distilled water the highest degrees of microleakage was observed in nanocomposite (Z-350XT), which is due to the nature of the porous zirconia and silica nanoclusters, as well as the presence of small non-agglomerated nanoclusters in Z-350XT, increasing the ratio of surface area to volume.^[38] Therefore, a wider area of hydrophilic silane is available to absorb water and this leads to accumulation of fluid in the space around filler-polymer interface. The absorbed water will not only diffuse within the polymer matrix but also will largely spread through filler-matrix interface and within the nanocomposite micro-bubbles. Curtis *et al.* in their study have reported that the size and morphology of filler particles affect water absorption and the mechanical properties. More surface area-to-volume ratio of fillers in nanofilled materials leads to an increase in water absorption and thus destroying the filler/matrix interface.^[39] The results reported by Ilie and Hickel were consistent with those of the current study, showing that during long-term storage in water and saliva nanocomposites undergo more degradation and deterioration compared to microhybride composite resins.^[40]

CONCLUSION

Despite the limitations of this *in vitro* study, the findings suggest that dental composite resins are not equally affected by artificial aging and restorative materials do not completely seal tooth-composite interface, although silorane-based composite resin is less significantly affected by the aging process. In 24-h, nanocomposite resin exhibited microleakage similar to silorane-based composite resin, but storage in water dramatically increased its microleakage.

However, further studies with longer storage times are needed to confirm these findings.

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