

## Microleakage Evaluation of a Silorane-Based and Methacrylate-Based Packable and Nanofill Posterior Composites (in vitro comparative study)

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### Key words

silorane, Filtek™ P90, Filtek™ P60, microleakage.

### Abstract

This study compared in vitro the microleakage of a new low shrink silorane-based posterior composite (Filtek™ P90) and two methacrylate-based composites: a packable posterior composite (Filtek™ P60) and a nanofill composite (Filtek™ Supreme XT) through dye penetration test. Thirty sound human upper premolars were used in this study. Standardized class V cavities were prepared at the buccal surface of each tooth. The teeth were then divided into three groups of ten teeth each: (Group 1: restored with Filtek™ P90, Group 2: restored with Filtek™ P60, and Group 3: restored with Filtek™ Supreme XT). Each composite system was used according to the manufacturer's instructions with their corresponding adhesive systems. The teeth were then thermocycled, immersed in 1% methylene blue dye for 24 hours at room temperature, embedded in auto-polymerizing acrylic resin and sectioned longitudinally bucco-lingually. Microleakage was evaluated by assessing the linear dye penetration at the tooth/restoration interface occlusally and gingivally. The highest microleakage score occlusally or gingivally was recorded and the results were analyzed statistically using SPSS version 13. The results of this study showed that the silorane-based posterior composite Filtek™ P90 showed significantly less microleakage than the methacrylate-based packable composite (Filtek™ P60) and the nano-filled composite (Filtek™ Supreme XT) when the tooth-restoration interface is located in enamel.

### Introduction

Resin composite materials have improved greatly since their introduction and there is a general shifting away from amalgams toward composite resins<sup>(1)</sup>. Although composites are now the material of choice for most restorations, their polymerization shrinkage remains a problem<sup>(2)</sup>. Composites consist of fillers embedded in a chemically-reactive organic resin matrix. Polymerization shrinkage is an intrinsic property of the resin matrix. Upon curing,

the single resin molecules move towards each other and are linked by chemical bonds to form a polymer network. This reaction leads to a significant volume contraction. It is striking, that during these decades of improvement, polymerization shrinkage was only incrementally reduced to a somewhat lower level<sup>(3)</sup>. Polymerization shrinkage and the resulting shrinkage stress, lead to microleakage which is among the major factors for composite material failures in the oral environment. Moreover, shrinkage stress can lead to tooth deformation, enamel

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cracks and stress-induced post-operative sensitivity<sup>(3)</sup>. Microleakage is defined as the clinically undetectable passage of bacteria, fluids, molecules, or ions between a cavity wall and the restorative material applied to it<sup>(4)</sup>. Microleakage at the tooth/restoration interface is considered a major factor influencing the longevity of dental restorations. It may lead to staining at the margins of the restoration, recurrent caries at the tooth/restoration interface, hypersensitivity of the restored teeth, and the development of pulpal pathology<sup>(5)</sup>. Furthermore, the differences in the coefficients of thermal expansion of the tooth and the resin composite can lead to different volumetric changes in the resin and the tooth structure during temperature changes<sup>(6)</sup>. The different volumetric changes directly affect microleakage<sup>(7)</sup>.

Improvements on the composite side were achieved, to a great extent, by optimizing the fillers, while the chemistry behind the organic resin matrix remained essentially the same since the pioneering work of R. L. Bowen in the 1960s. Practically all composites employ dimethacrylates such as TEGDMA, Bis-GMA or UDMA, which are radically polymerized as the primary resin. So the main strategy to reduce shrinkage was focused on increasing the filler load, thereby reducing the proportion of the methacrylate resin. Since the shrinkage is caused by the resin, the lower the proportion of resin in a composite, the lower the shrinkage will be<sup>(3)</sup>. The advent of nanotechnology in the field of dentistry is based on production of nanocomposites by improving the filler technology of submicron particle size, modification of organic matrix and silane coupling agent<sup>(8)</sup>. The nanotechnology is aimed to improve the physical and mechanical properties of the composite restoratives. Moreover, inclusion of smaller filler particles as nano-size in the final formulation of the composites results in reduction of composite's shrinkage and improving their total mechanical properties<sup>(9)</sup>. However, the shrinkage intrinsic to the methacrylate resin has remained a major challenge. Therefore, exchanging the resin seems the most promising pathway to solve the shrinkage

problem<sup>(3)</sup>. Siloranes are a totally new class of compounds for the use in dentistry. The name silorane derives from its chemical building blocks siloxanes and oxiranes. The combination of the two chemical building blocks of siloxanes and oxiranes provides the biocompatible, hydrophobic and low-shrinking silorane base of Filtek™ P90 Low Shrink Posterior Restorative recently introduced by 3M ESPE. This innovative resin matrix represents the major difference of Filtek™ P90 restorative compared to conventional methacrylates<sup>(10)</sup>. The polymerization process of Filtek™ P90 restorative occurs via a cationic ring-opening reaction which results in a lower polymerization contraction, compared to the methacrylate-based resins which polymerize via a radical addition reaction of their double bonds. The ring-opening step in the polymerization of the silorane resin significantly reduces the amount of polymerization shrinkage which occurs in the curing process. During the polymerization process, molecules have to approach their "neighbors" to form chemical bonds. This process results in a loss of volume, namely polymerization shrinkage. In contrast to the linear-reactive groups of methacrylates, the ring-opening chemistry of the siloranes starts with the cleavage and opening of the ring systems. This process gains space and counteracts the loss of volume which occurs in the subsequent step, when the chemical bonds are formed. In total, the ring-opening polymerization process yields a reduced volumetric shrinkage as claimed by the manufacturer<sup>(10)</sup>. This study was conducted with aim of comparing in vitro the microleakage of a new low shrink silorane-based posterior composite (Filtek™ P90) and a methacrylate-based packable posterior composite (Filtek™ P60) and a nanofill composite (Filtek™ Supreme XT) through dye penetration test.

## Material and Method

### Sample Selection

Thirty sound human upper premolars, extracted for the purpose of orthodontic treatment, were collected for use in this

study. The teeth were cleaned with pumice and carefully rinsed with water to remove the residual debris. Then the teeth were examined with a magnifying lens and light from a light curing unit for the presence of cracks. Only intact teeth free of defects and of comparable size were selected and stored in distilled water at room temperature.

### Cavity Preparation

Standardized class V cavities were prepared at the buccal surface of each tooth with the gingival margin 1 mm occlusal to the cemento-enamel junction. This means that all cavo-surface line angles were prepared in enamel. A template was used to trace the outline of the cavity on the buccal surface with 3 mm width, 2 mm height and 2 mm depth. The depth of the cavity was calibrated using a periodontal probe. Cavity preparation was done using a tungsten carbide fissure bur no. 330 (Komet, Germany) in a high-speed handpiece (W&H, Austria) mounted on the vertical arm of a surveyor with copious water spray. The cavo-surface line angles were finished with a stainless steel fissure bur no. 330 (Komet, Germany) in a low-speed handpiece (W&H, Austria) to produce a butt joint cavo-surface margins. A new bur was used for each five preparations. Non-beveled butt joint cavo-surface line angles were prepared all around in order to avoid the risk of reducing or entirely removing the thin enamel layer cervically<sup>(11)</sup>.

### Sample Grouping

The teeth were divided into three groups of ten teeth each according to the type of composite used to restore the cavities:

**-Group 1:** restored with a low shrink silorane-based posterior composite (Filtek™ P90) (3M ESPE, USA). Filtek™ P90 is a light-curing, radioopaque, silorane-based composite for the posterior area. It contains a hydrophobic resin matrix. It contains 55% volume (76% weight) inorganic fillers with a particle size between 0.1 and 2 μm<sup>(12)</sup>.

**-Group 2:** restored with a packable methacrylate-based posterior composite (Filtek™ P60) (3M ESPE, USA).

Filtek™ P60 restorative material is a visible-light activated, radiopaque, restorative composite, designed for use in posterior restorations. The filler in Filtek™ P60 restorative is zirconia/silica. The inorganic filler loading is 61% by volume (without silane treatment) with a particle size range of 0.01 to 3.5 μm. It contains BIS-GMA, UDMA and BIS-EMA resins<sup>(13)</sup>.

**-Group 3:** restored with a nanofill methacrylate-based composite (Filtek™ Supreme XT) (3M ESPE, USA).

Filtek™ Supreme XT universal material is a visible-light activated, restorative composite designed for use in anterior and posterior restorations. The fillers are a combination of aggregated zirconia/silica cluster filler with an average cluster particle size of 0.6 to 1.4 μm with primary particle size of 5-20 nm and non-agglomerated/non-agglomerated 20 nm silica filler. The inorganic filler loading is 59.5% by volume (78.5% by weight). It contains BIS-GMA, UDMA, TEGDMA, and BIS-EMA resins<sup>(14)</sup>.

### Restorative Procedure

Each composite system was used according to the manufacturer's instructions with their corresponding adhesive systems. Shade A3 was used for each composite type. Filtek™ P90 composite resin comes with a specially developed system adhesive (P90 System Adhesive, 3M ESPE AG, Germany) which consists of a self-etch primer and a bond. The primer was applied to the entire surfaces of the cavity with a disposable brush and massaged over the entire area for 15 seconds, then air dried gently, and light cured for 10 seconds with a Coltolex halogen light curing unit (Coltene Whaladent, France). Then the bond was applied to the entire surface of the cavity with a disposable brush, air dried gently, and light cured for 10 seconds as for the primer. Then Filtek™ P90 composite resin was applied to the cavity of each tooth in Group 1 with a plastic instrument and light

cured for 40 seconds according to the manufacturer's instructions<sup>(12)</sup>. For Filtek™ P60 and Filtek™ Supreme XT, Adper™ Single Bond 2 Adhesive (3M ESPE, USA) was used. It is a moist bonding adhesive containing 10%, 5 nm colloidal filler. The use of Adper™ Single Bond 2 Adhesive was preceded by acid etching of enamel and dentin as a part of the procedure. Phosphoric acid gel (META Biomed Co., Ltd, Korea) was applied to enamel and dentin for 15 seconds, rinsed for 10 seconds. Excess water was removed using a cotton pellet. The surface should appear glistening without pooling of water. Then Adper™ Single Bond 2 Adhesive was applied to the etched enamel and dentin using a fully saturated disposable brush for 15 seconds with gentle agitation, followed by gentle air thinning for five seconds to evaporate solvents, and light cured for 10 seconds<sup>(15)</sup>. Then Filtek™ P60 and Filtek™ Supreme XT composite resins were applied to the cavities of Group 1 and Group 2 respectively with a plastic instrument and light cured for 20 seconds according to the manufacturer's instructions<sup>(13,14)</sup>.

### Thermocycling

Thermocycling was done to simulate temperature changes in the oral cavity that might result in changes in the microspace between the tooth and the restoration. This was done by cycling the teeth between two water baths: one of the water baths maintained at 5°C and the other at 55°C, with a dwell time of 30 seconds. The number of cycles was 10 cycles assuming ten cycles of hot and cold drinking per day<sup>(16)</sup>.

### Dye Penetration Test

After thermocycling, the teeth were dried and their apices were blocked with auto-polymerizing acrylic resin to prevent dye penetration through the apical foramen. Then the teeth were coated with two layers of nail varnish to within approximately 2 mm of the tooth/restoration interface to prevent dye penetration from anywhere except via the tooth/restoration interface.

The teeth were then immersed in 1% methylene blue dye for 24 hours at room temperature.

### Sectioning

After dye penetration, the teeth were rinsed well under running water, dried, and embedded in blocks of auto-polymerizing acrylic resin. Then the teeth were sectioned longitudinally buccolingually through the cusps' tips using a diamond wheel mounted in a sectioning machine (Accutom) with water cooling.

### Microleakage Measurement

Microleakage was evaluated by assessing the linear dye penetration at the tooth/restoration interface occlusally and gingivally according to the scoring system used by Radhica et al. (2010)<sup>(17)</sup> by examining the sections of the teeth using a dissecting microscope (Wild, Heerbrugg, Switzerland) under a magnification of x4 as follows:

- Score 0 = no dye penetration.
- Score 1 = dye penetration into enamel.
- Score 2 = dye penetration beyond the dentino-enamel junction.
- Score 3 = dye penetration into the axial wall.

The highest microleakage score occlusally or gingivally was recorded and the results were analyzed statistically using SPSS version 13.

### Results

The descriptive statistics (mean, standard deviation, minimum and maximum) of dye penetration scores at the tooth/restoration interface of the different groups are shown in (Table 1). From this table, it can be seen that Filtek™ P60 composite showed the highest microleakage scores, followed by Filtek™ supreme XT composite, while Filtek™ P90 showed no dye penetration at all (score 0). Comparison of dye penetration scores at the tooth/restoration interface of the different types of

composite by one-way ANOVA test revealed a statistically highly significant difference among the different groups as shown in (Table 2). Further comparisons between groups by LSD test revealed a statistically highly significant difference in dye penetration scores between the teeth restored with Filtek™ P90 and those restored with Filtek™ P60 and Filtek™ Supreme XT, and a statistically significant difference in dye penetration scores between the teeth restored with Filtek™ P60 and those restored with Filtek™ Supreme XT shown in (Table 3). Figures 1, 2, and 3 show longitudinal sections of teeth restored with Filtek™ P90, Filtek™ P60, and Filtek™ Supreme XT, respectively.

## Discussion

Microleakage evaluation is the most common method of assessing the sealing efficiency of a restorative material<sup>(18)</sup>. In this study, the highly significant decrease in microleakage of the cavities restored with the Filtek™ P90 composite resin as compared with the Filtek™ P60 and Filtek™ Supreme XT composite resins could be attributed to the inherent ring opening polymerization of the silorane monomers in Filtek™ P90 composite resin which can compensate the volume reduction as the molecules come closer to each other compared to the radical polymerization of the methacrylate-based Filtek™ P60 and Filtek™ Supreme XT composite resins, which is linear polymerization, manifested as a reduction in polymerization shrinkage stress at the tooth/restoration interface. It is also hypothesized that since silorane technology provides lower polymerization shrinkage and related polymerization stress than methacrylate-based composite resins, it should be able to withstand thermocycling fatigue at the tooth/restoration interface better than the methacrylate-based composite resins<sup>(19)</sup>. This finding is in agreement with the results of Palin et al. (2005)<sup>(20)</sup>, Yamazaki et al.

(2006)<sup>(2)</sup>, and Bagis et al. (2009)<sup>(21)</sup>, who all reported that silorane-based resin

composite had significantly lower polymerization contraction than the methacrylate-based composite resins investigated. This would appear to indicate that ring-opening has in fact taken place with a concomitant contraction that is relatively small. However, this finding disagrees with the results of Ernst et al. (2009)<sup>(18)</sup>, who found that the microleakage of teeth restored with silorane was similar to others restored with the methacrylate-based composite Tetric Ceram and Clearfil SE Bond. The contrary in results could be attributed to the difference in the type of adhesive system used which is an all-in-one (7th generation) experimental bond of silorane previously produced by the company, while in this study, the new bond produced with silorane which is two-steps, two-components bond (6th generation) was used. On the other hand, the high microleakage scores seen with the Filtek™ P60 composite resin as compared with the Filtek™ Supreme XT composite resin could be attributed to the increase in the amount of filler particles and a consequent reduction in viscosity of the Filtek™ P60 composite resin, leading to an inadequate adaptation to the enamel walls<sup>(22)</sup>. The packable composites have insufficient matrix available for wetting the cavity wall and melting of the subsequent layers leading to formation of voids<sup>(13)</sup>. Other factors which might contribute to the high leakage scores of Filtek™ P60 and Filtek™ Supreme XT composite resins could be:

1. The difference between the coefficient of thermal expansion of the composite resin and that of the tooth structure due to high filler content<sup>(23)</sup>, and this further decreased the marginal adaptation when the specimens were thermocycled<sup>(17)</sup>.

This is in agreement with the results of Al-Boni and Raja (2010)<sup>(19)</sup>, who found that the Filtek™ P60 composite resin was the most affected by thermocycling. They reported that the thermal stresses encountered following thermocycling resulted in failure of the adhesive system at the interface of the tooth and restoration and may have contributed to the increase in microleakage.

2. In order to obtain a good seal, the adhesive monomers have to penetrate into the collagen network. After etching, if this network is dehydrated, the collagen will collapse and will impede the penetration of monomers and the formation of a good hybrid layer. The wet bonding technique implies that the dentin surface must be moist when applying the adhesive in order to promote the diffusion of the polymerizable monomers. After the acid etching, water fills the spaces in the collagen network. Primers must bring enough monomers to fill these spaces in the network and take water's place. To do so, the primer is combined to a solvent, such as acetone or ethanol, which help to remove water from the collagen network. The Adper™ Single Bond 2 Adhesive used in this study with Filtek™ P60 and Filtek™ Supreme XT composite resins contains water. This extra water competes

with the alcohol solvent, preventing its efficient penetration through the collagen network, leading to the formation of voids<sup>(24)</sup>. On the other hand, the P90 System Adhesive used with Filtek™ P90 composite resin relies on a self-etch primer which simultaneously decalcifies the inorganic component and infiltrates the collagen fibers at the same time, thus minimizing the potential for voids<sup>(25,26)</sup>.

### Conclusion

The silorane-based posterior composite Filtek™ P90 showed significantly less microleakage than the methacrylate-based packable composite (Filtek™ P60) and the nano-filled composite (Filtek™ Supreme XT) when the tooth-restoration interface is located in enamel.

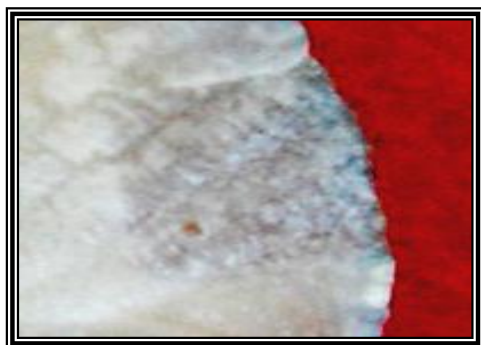


Fig.(1):- Longitudinal section of a tooth restored with Filtek™ P90 showing no dye penetration occlusally and gingivally(score 0).

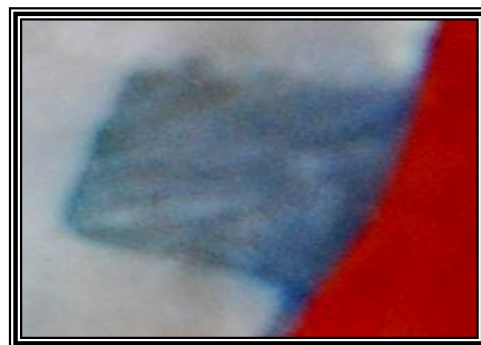


Fig.(2):- Longitudinal section of a tooth restored with Filtek™ P60 showing dye penetration up to the axial wall (score 3).

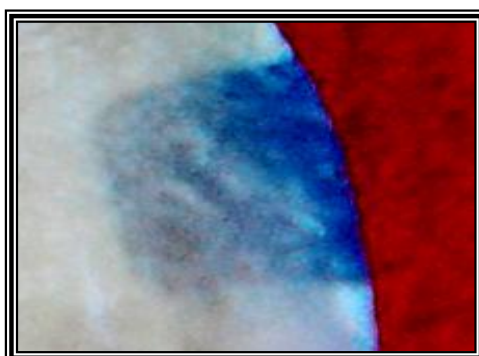


Fig.(3):- Longitudinal section of a tooth restored with Filtek™ Supreme XT showing dye penetration beyond the dentino-enamel junction occlusally and gingivally (score 2).

Table (1):- Descriptive statistics of dye penetration scores at the occlusal and gingival walls of the different groups.

| Groups  | N  | Mean | S.D    | Minimum | Maximum |
|---------|----|------|--------|---------|---------|
| P90     | 10 | .100 | .31623 | .00     | 1.00    |
| P60     | 10 | 2.70 | .48305 | 2.00    | 3.00    |
| Supreme | 10 | 2.30 | .48305 | 2.00    | 3.00    |

Table(2):- One-way ANOVA test for comparison of dye penetration scores at the tooth/restoration interface of the different types of composite.

|                | Sum of squares | df | Mean Square | F      | Sig.  |
|----------------|----------------|----|-------------|--------|-------|
| Between Groups | 39.200         | 2  | 19.600      | 103.76 | .000  |
| Within Groups  | 5.100          | 27 | 0.198       |        | (HS)* |
| Total          | 44.300         | 29 |             |        |       |

\*(HS): Highly significant (p≤0.01)

Table (3):- LSD test for comparison of dye penetration scores at the tooth/restoration interface of the different types of composite.

| Groups          | Sig.       |
|-----------------|------------|
| P90 * P60       | .000 (HS)* |
| P90* Supreme XT | .000 (HS)* |
| P60* Supreme XT | .05 (S)**  |

\*(HS): Highly significant (p≤0.01)

\*\* (S): Significant (p≤0.05)

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