

# Comparative evaluation of microleakage in class II cavities restored with Ceram X and Filtek P-90: An *in vitro* study

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## Abstract

**Context:** Polymerization shrinkage in composite resins is responsible for microleakage. Methacrylate-based composite resins have linear reactive groups resulting in high polymerization shrinkage. A recently introduced composite resin Filtek P90 is based on siloxanes and oxiranes which polymerize by cationic “ring opening” polymerization resulting in reduced polymerization shrinkage. **Objectives:** Aim of this study was to compare microleakage in class II cavities restored with a nanoceramic restorative (Ceram X) and a silorane composite (Filtek P90). **Materials and Methods:** Standardized class II box type cavities were prepared on mesial (Groups Ia and IIa) and distal (Groups Ib and IIb) surfaces of twenty extracted permanent molar teeth with gingival floor ending 1 mm coronal and apical to the cemento-enamel junction, respectively. The teeth in Group Ia and Ib were restored with Ceram X and Group IIa and IIb with Filtek P90. The specimens were thermocycled and microleakage evaluated. **Statistical Analysis Used:** The data were statistically analyzed using Wilcoxon Signed-Rank test at the 0.05 level of significance. **Results:** Mean microleakage score of group Ia and Ib was  $1 \pm 2.260$  and  $2.8 \pm 1.229$ , respectively. And that of group IIa and IIb was  $0.2 \pm .869$  and  $0.3 \pm .588$ , respectively. When groups I and II were compared, results were statistically significant ( $P < 0.05$ ). **Conclusion:** It was concluded that silorane-based composite may be a better substitute for methacrylate-based composites

**Keywords:** Cationic ring-opening monomers, oblique incremental technique, polymerization shrinkage, resin-based composite, silorane

## Introduction

Dental composite restorative resins have been available for nearly 50 years. Their specific use in posterior teeth was introduced in early 1980s and has become increasingly popular in restorative dentistry.<sup>[1]</sup> In recent years, the demand for posterior resin composite restorations has dramatically increased because of their ability to match tooth color, absence of mercury, thermally nonconductive, biocompatibility, and ability to bond to tooth structure.<sup>[2,3]</sup> Although the mechanical properties and abrasion resistance of resin-based composites have improved considerably over the years, the placement of posterior resin-based restoration remains very technique sensitive.<sup>[4]</sup>

Modern posterior resin composites undergo 2.6 to 7.1%

volumetric contraction during polymerization.<sup>[3,5-8]</sup> This shrinkage can result in microleakage and its ensuing sequelae.

Polymerization shrinkage has been considered as the most important factor responsible for marginal gaps around restorations resulting in microleakage. Direct Class II resin-based composite restorations can be done to an acceptable standard if the gingival margin is in sound enamel, but the quality of the margin of an adhesive restoration located below the cemento-enamel junction (CEJ) is questionable.<sup>[4]</sup> Since the bond with dentin is weaker, the polymerization shrinkage of the resin-based composite may lead to separation of the resin from the preparation wall and the formation of gaps.<sup>[4]</sup> Resins shrink during polymerization because the monomer units of the polymer are located closer to one another than they are in the original monomer.<sup>[9]</sup>

To reduce shrinkage stress effects, different restorative techniques have also been suggested. Among these are different types of sandwich restorations, different incremental placement techniques of the resin composite, and different light-curing regimens.<sup>[10]</sup> Various incremental techniques have been used for the placement of composite resin restorations like occlusogingival layering,<sup>[11]</sup> oblique layering,<sup>[11-13]</sup> faciolingual layering,<sup>[11]</sup> and centripetal placement<sup>[13]</sup> technique but none has been able to eliminate microgap formation at gingival margin. Among these, the oblique layering technique was found to show less microleakage than bulk or other incremental techniques as demonstrated by Neiva IF *et al.*<sup>[7]</sup> in 1998, Duarte S and Saad<sup>[13]</sup> in 2008, and Eakle WS and Ito<sup>[14]</sup> in 1990. However, none

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	DOI: 10.4103/0976-237X.94539

of the techniques has been able to eliminate the microgap formation completely.

Certain photoactivation protocols have been developed which serve as alternatives to high irradiance continuous curing, i.e., ramped cure,<sup>[15,16]</sup> step cure,<sup>[15,16]</sup> and pulse delay.<sup>[15-18]</sup> This slow polymerization reaction influences flow characteristics and may be useful in moderating the development of shrinkage stress and improving marginal adaptation.<sup>[19]</sup> But recent studies have reported a trend toward higher softening in ethanol for polymers formed by pulse-delay curing, suggesting that a polymer with a linear structure and reduced cross-linking density was formed.<sup>[18]</sup>

So, the factor responsible for microleakage is the inherent shrinkage in the organic matrix. Methacrylate-based composite resins have linear reactive groups and polymerize by addition reaction which results in high polymerization shrinkage. Use of organically modified ceramic nanoparticles and nanofillers combined with conventional glass fillers in Ceram X has improved the natural esthetics, reduced the monomer release, but polymerization shrinkage is still high, i.e., about 2.3% according to the manufacturers.<sup>[20]</sup>

Recently, a new composite resin Filtek P90 has been developed. It uses blocks of siloxanes and oxiranes to provide a biocompatible, hydrophobic, low-shrinking silorane as base. In these resins, polymerization takes place by cationic “ring – opening” mechanism resulting in minimal polymerization shrinkage of less than 1%.<sup>[21]</sup>

The aim of this *in vitro* study was to compare the microleakage in class II cavities restored with a nanoceramic restorative (Ceram X) and a silorane composite (Filtek P90) with gingival margin above and below CEJ. The null hypothesis tested was that the placement of gingival margin and the monomer systems would have no effect on the microleakage.

## Materials and Methods

Twenty noncarious extracted human mandibular molars were selected. The teeth were cleaned of calculus, soft tissue, and debris with hand instrumentation and stored in isotonic saline solution at 4 °C for not more than one month after extraction. Each specimen provided two surfaces for preparation, mesial and distal. To simulate clinical situation during restoration placement, a “restoration template” of size 3.5” x 4.5” was fabricated [Figure 1]. Two molars were embedded in dental stone to the level of CEJ, approximately 11 mm apart. Test specimen was embedded between these two teeth in polyvinyl siloxane impression material. Class II box type cavities were prepared on both mesial and distal surfaces of each tooth using a new straight fissured diamond bur (No.010) in a high-speed air water-cooled handpiece. The dimensions of the preparation were 4 mm (buccolingual width) with a pulpal depth of 2 mm. The gingival floor on

mesial side was prepared 1 mm below the CEJ and that on distal side was prepared 1 mm above the CEJ [Figure 2]. Each bur was used to cut four preparations only. Cavities were restored with an oblique incremental technique using the standard Palodent-contoured sectional matrix.

The teeth were divided into two groups according to the type of composite resin used to restore the teeth:

Group I: those restored with Ceram X

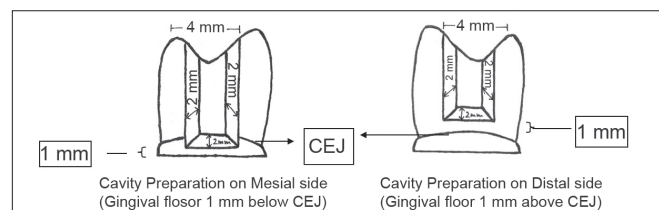
Group II: those restored with Filtek P90

In Group I, 36% phosphoric acid was applied to the cavity surfaces dentin for 15 seconds. The gel was removed by rinsing the cavity with water for 30 seconds. Prime and Bond<sup>®</sup> NT™ adhesive was dispensed directly on a fresh applicator tip and immediately ample amounts of Prime and Bond NT was applied twice to thoroughly wet all the tooth surfaces for 20 seconds. The cavities were gently air dried for 5 seconds and light cured for 10 seconds with a light curing unit. Palodent matrix system was applied. Teeth were restored with Ceram X mono using oblique incremental technique in 2 mm increments as shown in Figure 3. Each increment was cured individually with light unit curing for 20 seconds.

In group II, self-etching primer (P90 System Adhesive Self Etching Primer) was applied with microbrush for 15 seconds followed by gentle air dispersion and light curing for 10 seconds using light curing unit. After that, P90 system



**Figure 1:** Restoration template of 3.5” x 4.5” depicting test specimen embedded between two molars in polyvinyl siloxane impression material with Palodent-contoured sectional matrix

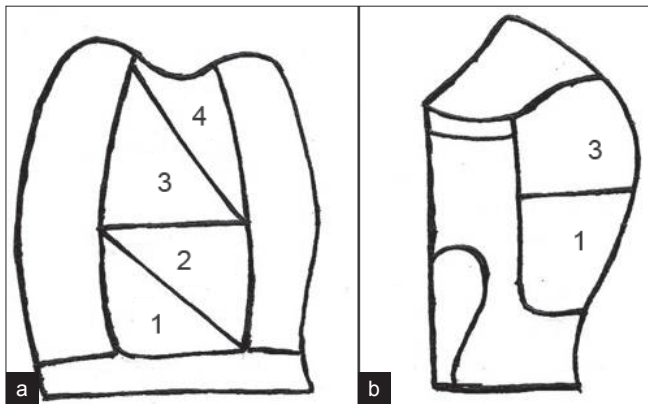


**Figure 2:** Dimensions of prepared class II box type cavities showing mesial and distal cavities with gingival floor 1 mm below and above CEJ, respectively

adhesive bond was applied to thoroughly wet all the cavity surfaces using a light brushing motion for 15 seconds followed by gentle air dispersion and light curing for 10 seconds with a light curing unit. Palodent matrix system was applied. P90 was placed immediately after the application and curing of P90 adhesive bond and was dispensed from the syringe into the prepared cavity by a flat plastic instrument using oblique incremental technique in 2 increments as shown in Figure 3. Each increment was cured individually with a light curing unit for 20 seconds.

After 24 hour storage in distilled water at 37°C, the restored teeth were subjected to artificial ageing by thermocycling. All the specimens were immersed alternatively in water baths at 5 ± 2°C and 55 ± 2°C for 1500 cycles with a dwell time 30 seconds and a transfer time of 15 seconds. In order to prevent dye penetration into the dentinal tubules and lateral canals, the apices were sealed with sticky wax and the teeth were coated with two layers of finger nail polish, except for an area approximately 1 mm around the margins of the restoration. The teeth were immersed in a 2% methylene blue aqueous solution for 24 hours. The sticky wax was removed following the dye exposure. Samples were then sectioned mesiodistally and dye penetration at enamel, dentin, and cementum margins [Figure 4] be scored under a stereomicroscope using the scores described below:

- 0- No dye penetration
- 1- Dye penetration extending to 1/3<sup>rd</sup> of the cervical wall
- 2- Dye penetration extending to 2/3<sup>rd</sup> of the cervical wall



**Figure 3:** Oblique incremental technique showing (a) cross-sectional view; (b) proximal view

- 3- Dye penetration into whole of the cervical wall
- 4- Dye penetration into the cervical wall and axial walls toward the pulp

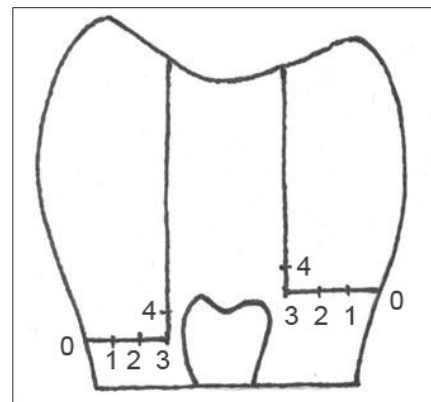
The data were statistically analyzed using Wilcoxon Signed-Rank test at the 0.05 level of significance.

### Results

The Table 1 shows the distribution of dye penetration scores. Mean microleakage score of group Ia and group Ib was 1 ± 2.260 and 2.8 ± 1.229, respectively. When group Ia and group Ib were compared, results were statistically significant ( $P < 0.05$ ). Mean microleakage score of group Ia and group IIb was .2 ± .869 and .3 ± .588, respectively. When group IIa and group IIb were compared, results were statistically insignificant ( $P > 0.05$ ) [Table 2]. When groups Ia and IIa were compared, results were significant ( $P < 0.05$ ). Comparison of group Ib and IIb was statistically significant ( $P < 0.05$ ) [Table 2]. However, when Group I and II were compared, results were statistically significant ( $P < 0.05$ ) [Table 2]. P90 showed less microleakage than Ceram X in cavities both above and below CEJ.

### Discussion

Recent advances in resin adhesives and restorative materials, as well as an increased demand for esthetics, have stimulated a great increase in the use of resin-based composites



**Figure 4:** Microleakage evaluation showing different microleakage scores

**Table 1: Microleakage scores**

Score	Above CEJ		Below CEJ	
	Group I <sub>a</sub>	Group II <sub>a</sub>	Group I <sub>b</sub>	Group II <sub>b</sub>
0	6	8	0	8
1	0	2	2	1
2	2	0	2	1
3	2	0	2	0
4	0	0	4	0
Mean ± S. D.	1 ± 2.260	0.2 ± 0.869	2.8 ± 1.229	0.3 ± 0.588

CEJ: Cementoenamel junction

in posterior teeth. Many commercially available dental composites are based on methacrylate chemistry. Because of the free radical polymerization of methacrylate-based composites, monomer molecules come closer to each other during the polymerization process, which results in polymerization shrinkage.<sup>[8]</sup> This volumetric shrinkage ranges from 2.6% to 7.1% and it develops stresses around the tooth restoration interface.<sup>[5-8]</sup> Polymerization contraction stress produces powerful forces that can result in cuspal deflection and separate the restoration from the tooth resulting in marginal microleakage.<sup>[8]</sup> Microleakage may cause hypersensitivity, recurrent caries, and pulpal pathosis and its sequelae.

Effective sealing of dentin tubules and, thus, the ability to withstand leakage forms a paramount factor in ensuring the longevity of a restoration.<sup>[4]</sup> Bonding to enamel is a relatively simple process, without major technical requirements or difficulties. Bonding to dentin, on the other hand, presents a much greater challenge due to the heterogenous nature of dentin.<sup>[22]</sup> So, the aim of this *in vitro* study was to compare the microleakage of two composites Ceram X and Filtek P90 with gingival margins placed above and below CEJ.

In the present study, twenty teeth in the age group of 35 to 45 years were taken. All the class II cavities had similar dimensions with no bevel so as to standardize the preparations. The teeth were divided into two groups, group I was restored with Ceram X and group II with Filtek P90. The gingival margins of the cavities were placed above (Groups Ia and IIa) and below (Groups Ib and IIb) CEJ. All the specimens were restored according to manufacturer's instructions using A2 shades of respective composites.

All tested materials were placed in 2 mm increments in oblique layering technique to minimize bridging between cavity walls and to reduce shrinkage stresses through the sequential use of small volumes of material. To simulate oral conditions, all the specimens were thermocycled.<sup>[23]</sup>

0.5% methylene blue dye was chosen as the agent of dye penetration to measure microleakage because it is simple, inexpensive, and does not require the use of complex laboratory equipment. Also, the particle size of this dye is less than the internal diameter of the dentinal tubules (1-4  $\mu$ m), so it is able to show dentin permeability.<sup>[24]</sup>

In this study, mean microleakage score in teeth restored with Ceram X (Groups Ia and Ib) was  $1 \pm 2.260$  and  $2.8 \pm 1.229$ , respectively. When Groups Ia and Ib were compared, results were statistically significant ( $P < 0.05$ ). The gingival/dentin margins showed significantly higher leakage than occlusal/enamel margins. The probable reason for this is that the bond strength to enamel is usually higher than bond strength to dentin. Dentin is less favorable bonding substrate due to its heterogenous structure.<sup>[4]</sup> However, enamel is a highly

**Table 2: Statistical analysis of group I and group II by wilcoxon signed ranks test**

Groups	Score	P value
I <sub>a</sub> - I <sub>b</sub>	0.008	<0.05
II <sub>a</sub> - II <sub>b</sub>	.914	>0.005
I <sub>a</sub> , II <sub>a</sub>	0.001	<0.05
I <sub>b</sub> - II <sub>b</sub>	0.000	<0.05
I - II	0.000	<0.05

< 0.05 – Statistically insignificant, >0.05 – Statistically significant

mineralized tissue composed of more than 90% (by volume) hydroxyapatite; dentin contains a substantial proportion of water and organic material, primarily Type I collagen. Dentin also contains a dense network of tubules that connect the pulp with the dentin-enamel junction.<sup>[22]</sup> The tubules may branch, particularly near the amelodentinal and cementodentinal junctions. Generally, branching of tubules are smaller and more numerous in root dentin than in crown dentin. Acid etching of the heterogenous dentin structure results in different surface chemistries and morphologies. Also, the orientation of dentin tubules can affect the formation of the hybrid layer. In areas with perpendicular tubule orientation, the hybrid layer was significantly thicker than areas with parallel tubule orientation.<sup>[4]</sup> Therefore, the dentin surface on the gingival floor of class II preparations may be a surface on which good hybrid layer formation is difficult. This could well contribute to the results of the present study.

Mean microleakage score of Filtek P90 (Groups IIa and IIb) was  $.2 \pm .869$  and  $.3 \pm .588$ , respectively. When Groups IIa and IIb were compared, results were statistically insignificant ( $P > 0.05$ ). Filtek P90 did not show statistically significant difference between the microleakage scores above and below CEJ. The probable reason for this may be explained by the fact that silorane system uses "ring opening polymerization instead of free – radical polymerization of dimethacrylate monomers. In this "living or dark" polymerization associated with the cationic mechanism, the reactive species do not become extinguished as quickly as the free radical contained within conventional methacrylate-based resin as reported by Palin WH *et al.*<sup>[25]</sup> in 2005; therefore, significantly lower polymerization shrinkage occurs irrespective of the location of the margins.

When Groups Ia was compared with IIa and Ib with IIb, Filtek P90 showed better sealing ability than Ceram X both above and below the CEJ. This is in agreement with studies of Bagis YH *et al.*<sup>[8]</sup> in 2009 who compared Silorane with nanohybrid composite (Grandio) and found Silorane-based microhybrid composite to have no microleakage. Papadogiannis D *et al.*<sup>[26]</sup> in 2009 also reported that Silorane material showed better behavior than dimethacrylate materials in setting shrinkage and marginal adaptation. The probable reason for this may be attributed to difference in filler loading<sup>[26-28]</sup> or filler size.<sup>[26,28]</sup> Ceram X is a nanohybrid resin composite. Nanostructures are used to produce composites with low

shrinkage, high wear resistance, and biocompatibility. Ceram X consists of organically modified ceramic nano-particles of 2.3 nm and nanofillers of 10 nm combined with conventional glass fillers of  $\sim 1 \mu\text{m}$ . Filler loading is 76% by weight and 57% by volume.<sup>[20]</sup> Filtek P90 restorative is filled with a combination of fine quartz particle and radiopaque yttrium fluoride classifying it as a microhybrid composite and has a filler loading of 76% and filler size of around  $0.1 \mu\text{m}$ .<sup>[21]</sup> Since the filler loading is almost same, it could not have affected the result obtained. However, the size of filler particle in Ceram X is less than that of P90, so Ceram X should show less polymerization shrinkage in accordance with various studies of Aw TC and Nicholls<sup>[28]</sup> in 1999, Papadogiannis D *et al.*<sup>[26]</sup> in 2009, and Satterthwaite JD *et al.*<sup>[27]</sup> in 2009. But the result obtained is contradictory to the above mentioned studies. The probable reason for this may be the difference in the matrix component in both the composites. So, the factor responsible for difference in microleakage in the above case may be the inherent shrinkage in the organic matrix. The polymer of most commercial dental composites is formed from dimethacrylates molecules whose polymerization reaction produces a densely cross-linked, but very heterogenous polymer network. Volumetric contraction is produced by the inherent density gain occurring when molecules previously existing at Vander Waal's force distances (0.3-0.4 nm) become linked through shorter covalent bonds (0.15 nm).<sup>[29]</sup> The reduction in free volume within the monomer structure as it transforms to a more densely packed polymer contributes to the overall contraction. The volumetric polymerization shrinkage of Ceram X is 1.7% and that of Filtek P90 is less than 1% according to the manufacturers. Silorane derives from the combination of its building blocks siloxanes and oxiranes. The network of Siloranes is generated by the cationic ring-opening polymerization of the cycloaliphatic oxirane moieties, which stand for their low shrinkage and low polymerization stress.<sup>[30]</sup>

However, when Groups I and II were compared, group II had better sealing ability than Group I and the results were statistically significant ( $P < 0.05$ ). Silorane-based composite showed less microleakage than Ceram X. Also, the marginal integrity and microleakage of silorane-based restorative systems are reported to be superior to methacrylate-based system by Palin WM *et al.*<sup>[25]</sup> in 2005. It may be due to the fact that P90 undergoes cationic curing while methacrylate-based composites undergo radical curing. The cationic cure in P90 starts with the initiation process of an acidic cation formed by the fragmentation of the photoinitiator which opens the oxirane ring and generates a new acidic center, the carbocation, which subsequently protonates the functional group of the oxirane molecule. After the addition to an oxirane monomer, the epoxy ring is opened to form a chain, or in the case of two- or multifunctional monomers, following molecular rearrangement, the positively charged species proceeds in three dimensions to form a tightly cross-linked network.<sup>[25,30]</sup> The decreased polymerization kinetics of the

oxirane compared with the methacrylate-based monomers generated a temporary excess of free volume that enhanced the mobility of the polymer chains within the system and, as a result, the polymerization efficiency of the cationic ring-opening monomers compared with the free radical species was increased.<sup>[25]</sup> So, silorane-based composites exhibit much less microleakage as compared with the nanohybrid composites, probably because of the difference in the matrix system.

However, clinical correlation and a larger sample size would be needed to assess the performance of the new material and to further establish the results.

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**How to cite this article:** Bogra P, Gupta S, Kumar S. Comparative evaluation of microleakage in class II cavities restored with Ceram X and Filtek P-90: An *in vitro* study. *Contemp Clin Dent* 2012;3:9-14.

**Source of Support:** Nil. **Conflict of Interest:** None declared.

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