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## Marginal leakage of class V cavities restored with silorane-based and methacrylate-based resin systems

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The aim of this study was to compare the microleakage of class V cavities restored by silorane resin with those restored by the methacrylate resin system. Sixty standardized class V cavities were prepared on premolars. The teeth were restored with Filtek™ Silorane and Silorane™ System Adhesive, Filtek™ Silorane and Adper™ Easy One and Filtek™ Supreme XT and Adper™ Easy One. The area and the depth of dye leakage were measured. Considering dye leakage area, the best results were achieved in group I (mean-M=0.114±0.083 mm<sup>2</sup>), then in group III (M=0.384±0.480 mm<sup>2</sup>) and finally in group II (M=5.389±6.587 mm<sup>2</sup>). Considering the depth of dye leakage, there was a significant difference between group I (mean rank-m.r.=1.23±0.44 mm) and II (1.98±0.83 mm), group II and III (1.23±0.40 mm). In this study silorane composite and its adhesive showed less microleakage than the methacrylate composite, and it showed compatibility only with its assigned adhesive. Clinical Significance: Less microleakage of silorane restorations could provide better and long lasting composite fillings.

**Keywords:** Silorane, Methacrylate composite resin, Microleakage, Adhesive system, Class V

### INTRODUCTION

Often, restorative materials and methods do not ensure a stable bond between the material and hard dental tissue<sup>1</sup>. Therefore, bonding of the composite materials to the hard dental tissues, as well as the longevity and stability of that bond, is of great importance for the ultimate success of the restoration<sup>2</sup>. The problem with composite restorative materials is related to the polymerization shrinking and the associated polymerizations stress, which causes micro cracks, recurrent caries, and post-operative sensitivity<sup>3-5</sup>. In recent years, a new class of composite materials has appeared, based on monomers with an opening ring<sup>6</sup>, named silorane. The name derives from their chemical composition, which is a combination of siloxane and oxirane<sup>7</sup>. Literature widely states their advantage over the composite materials based on methacrylate, due to the reduced amount of contraction during the polymerization, as well as good mechanical properties and an enhanced hydrophobicity needed for long term intraoral stability<sup>7,8</sup>. In order to ensure the best possible bonding between the silorane composites and hard dental tissues, a special adhesive has been developed especially for silorane. Silorane adhesives are made from the hydrophilic one step self-etching primer and the hydrophobic viscous bond coating resin<sup>9</sup>. The manufacturers' recommendation is that only silorane-compatible adhesives are to be used. However, since silorans are bonded with two-step adhesives, it is

arguable whether they can also be used with one-component adhesives, normally used for composite materials based on methacrylate, therefore facilitating and simplifying the work.

Marginal adaptation is an important concern in restorative dentistry, and it can be assessed in a laboratory by the degree of marginal leakage. Testing for marginal leakage by dye-penetration is historically the oldest and most frequently used method. The major disadvantage of the method is that samples are often damaged in the process of cutting, which can interfere with the results<sup>10-12</sup>. An ideal way of testing would be to remove an undamaged filling from the cavity and then observe the microleakage in its full extent. A so called 3-D view method of testing the bonds between the fillings and the hard dental tissue enables such observation, and consequently contributes to a better understanding of the circumstances in which marginal leakage occurs<sup>13-15</sup>.

The aim of this research is to evaluate the microleakage of the class-V fillings made of the silorane resin material compared with the Filtek™ Supreme XT methacrylate-based composite-material fillings. Also, the aim is to determine the compatibility of adhesives based on methacrylate and silorane resin.

### MATERIALS AND METHODS

Composite resins used in this study were Filtek™ Silorane (3M ESPE, St. Paul, MN, USA) and Filtek™ Supreme XT (3M ESPE). Filtek™ Silorane is composed 23% silorane resin which is the combination of two

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chemical building blocks siloxanes and oxiranes. The filler component is a combination of fine quartz particles and radiopaque yttrium fluoride. From the filler side, Filtek Silorane restorative is classified as a microhybrid composite. Filtek™ Supreme XT is a direct restorative nanocomposite. The resin system is the same reduced shrinkage resin as found in 3M ESPE's Filtek™ Z250. All the shades besides the translucent one contain a combination of a non-agglomerated, 20 nm nanosilica filler, and loosely bound agglomerated zirconia/silica nanocluster, consisting of agglomerates of primary zirconia/silica particles with size of 5–20 nm fillers and the filler loading by 78.5% weight (Table 1).

The research was performed on 30 intact human premolars with completely developed roots which had been extracted for orthodontic reasons. The teeth were kept in 1% solution of chloramine (Kemika, Zagreb, Croatia) at room temperature during the period between 1 and 6 months. The cavities were prepared by only one operator with a high speed air handpiece under water cooling with a specially designed diamond drill (#811 031 4.2ML, Diatech, SDI, Switzerland) in order to obtain uniform cavities. The shape of the cavities was conical with divergent walls and a flat bottom with dimensions 3×2×1.5 mm. Two cavities were made on each tooth. The cavities were made under water-cooling on the vestibular and the oral surfaces of the tooth so that the occlusal edge of the cavity ended in the enamel and the gingival one ended in cement, whereas part of the wall and the bottom of the cavity were in the dentin (ca. 2 mm deep). The same drill was used on no more than 12 cavities to avoid irregularity of the bur due to wear and tear, and to consequently keep the size and shape of the cavities reasonably constant.

Sample-teeth were randomly divided into three groups. The class V cavities were drilled on the buccal and lingual surfaces of each tooth. The enamel part

of the cavities were etched with 37%-orthophosphoric acid (Ivoclar Vivadent AG, Schaan, Liechtenstein) for 15 s, following manufacturer's instruction. After the etching, each cavity was rinsed with tap water for 15 s, and the excessive water was forced out of the cavity by two separate brisk jets of air so that the surface of the cavity remained wet.

The first experimental group of teeth (Group I) was restored with Filtek™ Silorane (3M ESPE, St. Paul, MN, USA) and Silorane System Adhesive (3M ESPE, Seefeld, Germany). Initially, the cavity was coated with a layer of Silorane System Adhesive by a self-etching primer (3M ESPE, Seefeld, Germany) for 20 s. The excessive primer was removed by a gentle flow of air and subsequently the light-polymerization was done for 10 s by Bluephase C8 Light Unit (Ivoclar Vivadent AG, Schaan, Liechtenstein). Then the cavity was coated with a layer of Silorane System Adhesive bond (3M ESPE, Seefeld, Germany) for 20 s. The bond was polymerized with the same device for 10 s after the removal of its excess by a gentle flow of air. After that, the cavities were restored with Filtek™ Silorane (A3) in one layer and the fillings were polymerized for 40 s.

The second experimental group (Group II) was restored with Filtek™ Silorane (3M ESPE, St. Paul, MN, USA) and Adper™ Easy One (3M ESPE, Seefeld, Germany). The cavity was coated with a single layer of Adper™ Easy One adhesive for 35 s. The excess of adhesive was removed by a gentle flow of air. Then the adhesive was light cured for 10 s. Filtek™ Silorane (A3) was then applied into the cavity in one layer and light cured for 40 s.

The third experimental group (Group III) was restored by Filtek™ Supreme XT (3M ESPE, St. Paul, MN, USA) and Adper™ Easy One (3M ESPE, Seefeld, Germany). The cavity was initially coated with the adhesive Adper™ Easy One for 35 s. The excess of adhesive was removed by a gentle flow of air. The

Table 1 Composition of used materials

Composites		
Filtek Supreme XT	Filtek Silorane	
Bis-GMA, Bis-EMA,UDMA, TEGDMA, non-agglomerated/non-aggregated nanosilica filler, zirconia/silica nanoclusters; 78.5 wt%	3,4-epoxycyclohexylethylcyclopolymethylsiloxane, bis-3,4-poxycyclohexylethylphenylmethylsilane, Silanized quartz; yttrium fluoride; 76 wt%	
Adhesives		
Adper Easy One	Silorane System Adhesive Primer	Silorane System Adhesive Bond
Bis-GMA, polyalkenoic acid co-polymer, dimethacrylates, phosphorylated methacrylates, HEMA, photoinitiators, ethanol, water, nanofiller particles	phosphorylated methacrylates, polyalkenoic acid co-polymer, Bis-GMA, HEMA, ethanol, water, photoinitiators, silane treated silica nanofiller	hydrophobic bifunctional methacrylate based monomer, acidic monomers, photoinitiators, silane-treated silica filler

Legend: Bis-GMA=bisphenol-A-glycidyl methacrylate; Bis-EMA=ethoxylated bisphenol-A-glycol dimethacrylate; UDMA=urethane dimethacrylate; TEGDMA= triethyleneglycol dimethacrylate; HEMA=2-hydroxyethyl methacrylate

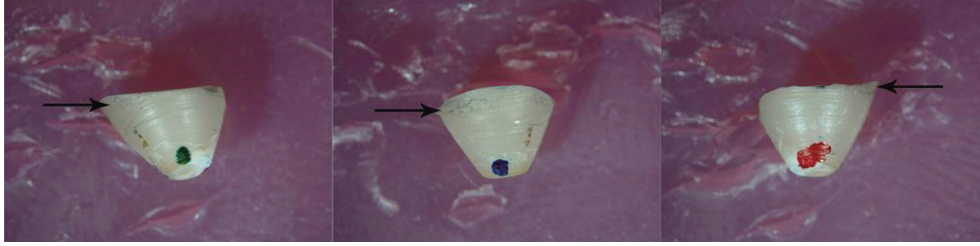


Fig. 1 Photograph of a sample of restoration done by Filtek Silorane and Silorane adhesive from three points of view. Red, green and blue dots enabled the differentiation of sides of filling. Dye surfaces are pointed with arrows.

cavity was then light-cured for 10 s. Filtek™ Supreme XT (A3) was then applied into the cavity in one layer and polymerized for 40 s. The surface of all cavities was treated with the Sof-Lex™ and Extra Thin/Coarse finishing disk (3M ESPE, St. Paul, MN, USA), mounted on a micro-electromotor, rotating at low rotation per min. The finishing disk was changed after every 20 cavities. Finally, all composite restorations were immersed into a saline solution (Natrii chloridi infundibile, 0.9% NaCl, Pliva, Zagreb, Croatia). After 24 h, restoration surfaces were polished, with a brush (Gebr. Brasseler GmbH & Co. KG, Lemgo, Germany) and the Proxyl® RDA 7-fine/fein polishing paste (Ivoclar Vivadent AG, Schaan, Liechtenstein). To keep the fillings overhang-free, all the fillings were polished. When the treatment of all cavities was completed, each group of specimens was subjected to 1,800 thermocycles at 5°C and 55°C with 60 s dwell time and 10 s transfer time.

The tip, as well as the entire root-surface of each dried tooth (apart from the very surface of the filling and the area up to 1–2 mm from the edge of the filling) was coated with red nail varnish (LCN GmbH, Eltville, Germany). After that, the teeth were immersed into black dye acid-resistant contrast-liquid (Ecoline-Royal Talens, Apeldoorn, Netherland) for 24 h. After the 24-h-period, the teeth were rinsed with water and immersed into freshly prepared 5%-nitric acid (HNO<sub>3</sub>) for the next 48 h. Forty eight hours later, the hard dental tissue had softened enough and the teeth were taken out from the acid and thoroughly rinsed with water. The intact fillings were then taken out from their cavities using a sharp excavator. The samples were analyzed by a stereoscopic microscope (Olympus Stereo SZX12, Tokyo, Japan) at 10× magnifications. Each filling was photographed by a digital still-camera mounted onto the microscope (Olympus, Tokyo, Japan). To get the entire contact-surface of the conical shape filling, each filling was photographed from three different profile views, each proximally 120° apart, and completing a full (360°) circle. Prior to that, the surface of each filling had been marked by pens of different colors (red, green and blue) at three equidistant points, to distinguish the sides of the fillings (Fig. 1).

The obtained photographs were processed by a

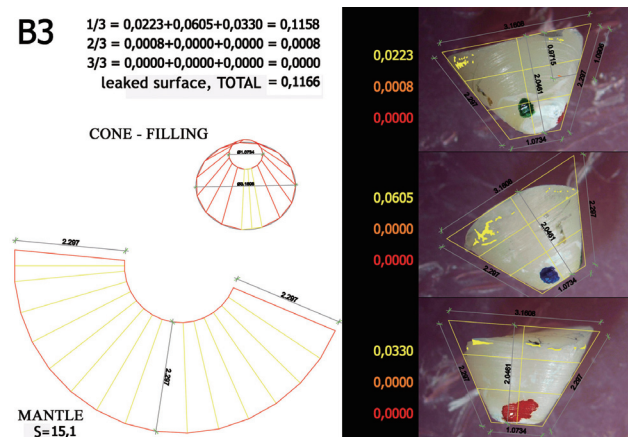


Fig. 2 Computer-processed sample restored with Filtek Silorane+Silorane adhesive.

computer program AutoCAD, version 2009 (AutoDesk Inc, San Rafael, CA, USA) and both the maximum depth, and the surface of dye-penetration were assessed for each specimen (Fig. 2). The results were analyzed using a non-parametric Kruskal-Wallis test and Mann-Whitney U-tests at a 95% significance level by using the SPSS program version 16.0.1 (IBM, Chicago, IL, USA).

## RESULTS

All tested samples, regardless of the material used or adhesive applied, had a dye-leakage between the fillings and the cavity-walls (Fig. 2). Six fillings from Group II had fallen out from their cavities during the process of thermocycling and they were statistically categorized as samples with total leakage. The mean values and standard deviation of the surface and the depth of dye-penetration are shown in Tables 2 and 3. The comparison between the three groups with regard to the entire surface dyed by the Kruskal-Wallis test showed a statistically significant difference ( $\chi^2=37.37$ ;  $df=2$ ;  $p=0.000$ ). The Mann-Whitney U-test showed statistically significant differences between the results of Group I (Filtek Silorane and Silorane System

Table 2 Descriptive statistics of dye-penetration surface expressed in square millimetres and differences between groups (Mann Whitney test)

	Number of samples	Mean value (mm <sup>2</sup> )	Standard deviation	Margin of error	Minimum	Maximum	Differences between tested groups		
							Filtek Silorane+ Silorane Adhesive	Filtek Silorane+ Adper Easy One	Filtek XT+ Adper Easy One
Filtek Silorane+ Silorane Adhesive	20	0.114	0.083	0.018	0.002	0.242	—	0.000*	0.001*
Filtek Silorane +Adper Easy One	20	5.389	6.587	1.473	0.040	15.1	0.000*	—	0.001*
Filtek Supreme+ Adper Easy One	20	0.384	0.480	0.107	0.107	2.07	0.001*	0.001*	—

\*Statistically significant ( $p < 0.05$ )

Table 3 Descriptive statistics of dye-penetration depth expressed in millimeters and differences between groups (Mann Whitney test)

	Number of samples	Mean value (mm)	Standard deviation	Margin of error	Minimum	Maximum	Differences between tested groups		
							Filtek Silorane+ Silorane Adhesive	Filtek Silorane+ Adper Easy One	Filtek XT+ Adper Easy One
Filtek Silorane+ Silorane Adhesive	20	1.233	0.445	0.099	0.615	1.978	—	0.000*	0.829
Filtek Silorane +Adper Easy One	20	1.976	0.827	0.018	1.780	2.110	0.000*	—	0.000*
Filtek Supreme+ Adper Easy One	20	1.234	0.399	0.089	0.455	1.807	0.829	0.000*	—

\*Statistically significant ( $p < 0.05$ )

Adhesive) and Group III (Filtek Supreme XT and Adper Easy One) ( $U=85.0$ ;  $Z=-3.111$ ;  $p=0.001$ ), between Group I and Group II (Filtek Silorane and Adper Easy One) ( $U=26.0$ ;  $Z=-4.714$ ;  $p=0.000$ ), and between Group II and Group III ( $U=72.0$ ;  $Z=-3.468$ ;  $p=0.001$ ). The statistical differences between the groups are shown in Tables 2 and 3. The order of the three groups, according to their Kruskal-Wallis test mean-rank, is the following: Group I (16.05), followed by Group III (29.85) and finally Group II (45.60). This means that the total of dyed surface is statistically far greater in those fillings made of silorane and an incompatible adhesive, compared to those made of silorane composite and a compatible adhesive, or the methacrylate-based composite and the compatible adhesive.

The comparison of the three groups, with regard to three different methods of treatment at the maximum depth of penetration by the Kruskal-Wallis test, showed a significant difference ( $\chi^2=36.29$ ;  $df=2$ ;  $p=0.000$ ). The Mann-Whitney U-test showed significant differences

between Group I and Group II ( $U=15.0$ ;  $Z=-5.01$ ;  $p=0.000$ ), and Group II and Group III ( $U=1.00$ ;  $Z=-5.39$ ;  $p=0.000$ ). However, the differences in the depth of penetration between Group I and III was not statistically significant ( $U=192.0$ ;  $Z=-0.25$ ;  $p=0.829$ ). The order of the three groups, according to their Kruskal-Wallis test mean-rank, is the following: the best ranked is Group I (20.85), followed by Group III (20.95), and finally Group II (49.70). This means that the maximum depth of dye-penetration in the teeth restored with either Filtek Silorane, or Filtek Supreme, both with the compatible adhesives, is significantly smaller than the maximum depth of dye-penetration in the teeth restored with Filtek Silorane in combination with an adhesive originally made for composites based on methacrylate.

## DISCUSSION

In the present study, marginal leakage of the composite materials based on a new chemical composition, silorane

monomers were compared with the traditionally used composite. Thereby, a 3D-results-reading method, which has advantages in comparison with other dye-penetration methods, where the samples and the surrounding tooth-tissues are being cut, was used. In the process of cutting, the samples may be damaged, rendering a precise assessment of dye-penetration depth doubtful, and reading of the surface of the dye-penetration impossible. The surface of dye-penetration is an important factor which reflects the quality of the bond between the fillings and the hard tooth-tissues, which is a more reliable parameter in the assessment of leakage than the one-dimensional assessment of the dye-penetration depth. A disadvantage of this method is manifested in potential errors which are due to inherent irregularities of the conical shape of the fillings, and those due to dealing with shapes of very small dimensions.

This research confirms the difference between the results obtained for depth and the surface of dye-penetration. A statistically significant difference was established between the fillings made with silorane system adhesive, and methacrylate-based resin adhesive system used in this study, with regard to the surface of the leakage. However there was not any statistical difference with regard to the other parameter, namely the dye-penetration depth for Groups I and III. This may explain the differences between the results in the present research, and the results of the research by Palin *et al.*<sup>16)</sup> who did not record any statistically significant difference of marginal leakage in silorane and Z100 (3M ESPE). Yet, they noticed such a difference for silorane and Filtek Z250 (3M ESPE). This shows that various traditionally used composite materials, due to a difference in their chemical compositions, may have a different marginal integrity, and therefore caution is advised when interpreting the comparable results. In a previous study on microleakage of Class II fillings by method of sample-cutting, Bagis *et al.*<sup>17)</sup> did not find that silorane fillings had any marginal leakages. The differences between the present study and Bagis' study could be attributed to different shapes of the cavities examined. Class V cavities pose a problem regarding the longevity of the fillings, due to a weaker adhesion of the restorative material to the dentine and to cement walls of the cavity, and due to the configuration factor<sup>18)</sup>, which could possibly explain the differences. The results of two different studies by Thalaker *et al.*<sup>19,20)</sup>, who tested the marginal integrity of the silorane and the methacrylate-based composite fillings by a scanning electron microscope are consistent with the results of our research. Thalaker *et al.*<sup>19,20)</sup> were able to confirm better marginal integrity in the silorane fillings initially after placing, after thermocycling, and after simultaneous thermocycling and exposure to mechanical stress. Among factors influencing micro-leakage are bond strengths of adhesive systems as well as polymerization shrinkage of composite resins. It was demonstrated that silorane adhesive system microtensile bond strength is comparable to that of the 6th generation self etch bond Clearfil SE bond

even after one year water storage<sup>21)</sup>. Considering the polymerization shrinkage, research have shown that silorane materials do have a lower shrinkage when compared to methacrylate based composites thus exhibiting lower polymerization stress (1.4–4.4 MPa for Filtek Silorane)<sup>22)</sup>. Also, thermocycling and food simulating liquids exposure affected silorane composite significantly less than conventional methacrylate-based composite material<sup>23-25)</sup>. Additionally, by the method of micro-Raman spectroscopy, it has been confirmed that the hybrid-layer, created by the silorane adhesive system, is of a comparable thickness to that created by the methacrylate-based adhesives<sup>26)</sup>. Similar results for thickness of the hybrid-layer were obtained by Duarte *et al.*<sup>9)</sup> by a scanning electron microscope. A corresponding hybrid-layer, with an added reduced contraction of the silorane composites, could explain better results in terms of micro-leakage obtained in the present study. A correlation between the level of polymerization shrinkage and microleakage was proved by Calheiros *et al.*<sup>27)</sup> who studied the composites with a lower level of polymerization contraction (with a higher content of pre-polymerized particles of the inorganic matrix).

The significant difference between Filtek™ Silorane and Filtek™ Supreme XT for the parameter of surface with leakage, in contrast to insignificant difference in the depth of the dye penetration, indicates that the penetration surface parameter describes the patterns of micro-leakage in a better way. The methods describing the surface of dye-penetration should become a standard when testing the adhesive characteristics of the materials for cavity-fillings.

Six samples from Group II (silorane composite and Adper Easy One) were found to have been completely detached from the tooth-tissues after thermocycling. Statistically, they were treated as samples with total leakage. The remainder of the samples showed a statistically significant difference with regard to both parameters (depth and the surface of dye-penetration), which proved that the bond between the methacrylate-based adhesives and silorane was insufficient. This is consistent with the research done by Duarte *et al.*<sup>9)</sup> who tested the microtensile bond strength and proved an insufficient bond of combination of methacrylate-based adhesives and silorane for dentine. Bearing in mind that siloranes are highly hydrophobic, a silorane system adhesive self-etch primer is rather hydrophilic, and ensures strong and durable adhesion to the tooth by overcoming the wetness of the dentine. Silorane system adhesive bond is optimized for wetting and adhering to the hydrophobic Filtek Silorane. Although the primer's mechanism of bonding to the hard dental tissues is similar to that of the methacrylate adhesives, the primer and the bond are expected to polymerize separately. Consequently, the bond between the tooth and the adhesive is established in the very first step and that makes them similar to the one-step self-etching adhesives<sup>28)</sup>. A poor bond between silorane composites in combination with adhesive for

methacrylate-based composites can, in addition to the chemical composition, be a result of insufficiently hydrophobic conditions present after the placement of adhesives. Unlike silorane bonding with methacrylate-based adhesives, silorane system adhesive can be used in combination with methacrylate-based composites<sup>29</sup>. The research of marginal leakage is a part of the laboratory testing of materials, but the longevity and the quality of fillings are influenced by a great number of factors which cannot be simulated under *in vitro* conditions. Therefore, clinical research is needed for their final evaluation. Nevertheless, based on lab-research data, it is possible to predict the marginal integrity of fillings in clinical conditions<sup>2</sup>.

### CONCLUSION

The results of this study showed that the Filtek™ Silorane had significantly less dye-leakage in comparison with Filtek™ Supreme observing the area of dye penetration. It was also shown that Filtek™ Silorane could only be used with its assigned adhesive system.

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