

Microleakage and Microtensile Bond Strength of Silorane-Based and Dimethacrylate-Based Restorative Systems

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Abstract— **OBJECTIVE:** To evaluate the microleakage and bond strengths (μ TBS) of silorane-based (SBC) and dimethacrylate-based (DBC) restorative systems. The null hypotheses are: 1) there is no difference in microleakage between the resin composites and the adhesive systems tested at the enamel and dentin margins; 2) there is no significant difference in μ TBS between the composite restorative systems using self-etch and etch-and-rinse adhesive versions. **METHODS AND MATERIALS:** *Microleakage:* Class V cavity preparations with cervical margin in dentin were performed on pristine extracted human molars and were randomly distributed among the following three groups: Group 1—DBC/etch-and-rinse adhesive system (Filtek™ Supreme Ultra/Adper™ Single Bond Plus [3M ESPE, www.3MESPE.com]); Group 2—SBC/self-etch adhesive (Filtek LS Low Shrink Posterior Restorative System/LS self-etch adhesive [3M ESPE]); Group 3—DBC/self-etch adhesive (Filtek Supreme Ultra/Adper Easy Bond Self-Etch [3M ESPE]). Restorations were thermocycled and immersed in 0.2% methylene blue dye for 24 hours. Samples were assessed visually under 10x magnification; *μ TBS:* Bond sticks (0.9 mm²) were prepared from each group and tested on a universal testing machine. **RESULTS:** The Kruskal-Wallis test revealed no significant difference in microleakage among the experimental groups at the enamel margin ($P = 0.191$). At the dentin margins, silorane/self-etch restorative system showed significantly less leakage than the dimethacrylate/etch-and-rinse restorative system ($P = 0.008$). Tukey's B rank order test showed that the dimethacrylate/etch-and-rinse restorative system presented the highest μ TBS. **CONCLUSIONS:** SBC/self-etch system showed less microleakage at the dentin margins, while DBC/etch-and-rinse system presented higher bond strength.

Keywords: adhesion, composite resins, dentin bonding agents

Dimethacrylate-based resin composites (DBC) have become an increasingly popular restorative material. This is due, in part, to their esthetics, long working time/command cure, and improved mechanical properties.¹ To date, however, DBCs universally suffer from one common drawback: polymerization shrinkage.² This well-documented phenomenon can lead to a compromise of marginal integrity, resulting in microleakage, postoperative sensitivity, marginal staining, recurrent caries, and, inevitably, restoration failure.^{2,3} To overcome these problems, research has focused on a number of solutions, including modifications to material composition such as alterations to the amount of filler and filler size and the use of high-molecular-weight monomers.^{4,5} Despite enormous improvements, however, the fundamental problem of polymerization shrinkage is related to the radical polymerization method of the methacrylate-based resin composites. Basically, all resin composites employ dimethacrylates, such as bisphenol A-glycidyl methacrylate (Bis-GMA), tetraethylene glycol dimethacrylate (TEGDMA), or urethane dimethacrylate (UDMA), as the primary resin. Through conversion of monomer molecules into a polymer network, van der Waals spaces are exchanged for shorter covalent bindings, resulting in a closer, tighter arrangement

of molecules, leading to a reduction in material volume.² Recently, a newer silorane-based composite (SBC) using a polymerization method termed “photo-ring-opening cationic polymerization” (ROCP) seems to have overcome this main drawback of polymerization shrinkage. Siloranes, so named for their component moieties, contain a siloxane backbone with four attached oxirane rings that open to form a polymer chain.⁵ The oxirane ring opening results in volumetric expansion that offsets the shrinkage resulting from monomer conversion into polymer. While still in their infancy as a clinical material, siloranes have already demonstrated mechanical properties on par with standard dimethacrylate-based composites,^{4,6} but with as little as under 1% polymerization shrinkage, about half that of the best current resin-based composites (RBCs).^{7,8} Also, a longer gelation time with a resulting reduced shrinkage strain has been reported.⁴ This translates to different polymerization rheology, allowing the material to better compensate for stresses during polymerization and improving marginal adaptation.⁷

Despite these positive aspects, an interesting caveat about SBC is its chemical incompatibility with universal methacrylate-based dentin bonding agents.⁶⁻⁸ Moreover, an SBC requires its own dedicated adhesive system for bonding to dentin and other DBC materials. The silorane-dedicated adhesive system

is available in a self-etch version, which, according to the manufacturer, was created to fulfill the criterion of simplification, with the reduced number of operative steps offered by self-etch adhesive systems. Although simplified adhesive versions appear to induce loss of effectiveness, good results have been reported for the two-step self-etch adhesives.⁹⁻¹³ Therefore, the aim of the current study was to evaluate microleakage in Class V resin restorations and determine the microtensile bond strength of SBC and DBC. The null hypothesis is twofold: 1) An SBC with its dedicated adhesive system does not perform better than a DBC with self- and etch-and-rinse adhesive versions in reducing microleakage; 2) there is no significant difference in μ TBS among SBC and DSC materials using self-etch and etch-and-rinse adhesive systems.

Materials and Methods

Thirty-six freshly extracted caries-free human third molars were selected for this study. After removal of both calculus deposits and soft tissues, the teeth were stored in a 0.1% thymol solution for a maximum of 3 months until use. Prior to the bonding experiments, the teeth were retrieved from the disinfectant solution and stored in distilled water, with daily changes of the latter for 2 weeks to remove the disinfectant. Approval to use human teeth was obtained from the Research Ethics Committee at the University of Western Ontario.

CAVITY DESIGN

Class V cavity preparations measuring 2 mm occluso-gingivally,

3 mm mesiodistally, and 1.5 mm in depth were prepared using a pear-shaped #245 bur (ISO 032, Brasseler, www.brasseler.com) under air-water cooling. The occlusal margin was located on the enamel, whereas the gingival margin was located in the dentin, 1 mm below the cemento-enamel junction (CEJ). Each carbide bur was discarded following preparation of each group of teeth. All measurements were confirmed using a calibrated periodontal probe to maintain uniformity. One operator did all cavity preparations to ensure a consistent calibrated cavity size and depth.

RESTORATIVE PROCEDURES

Teeth were then randomly assigned to three experimental groups (n = 12) according to the following restorative systems: Group 1—dimethacrylate-based resin composite/etch-and-rinse adhesive system (DBC-ER) (Filtek Supreme Ultra/Adper Single Bond Plus); Group 2—dimethacrylate-based resin composite/self-etch adhesive (DBC-SE) (Filtek Supreme Ultra/Adper Easy Bond Self-Etch); Group 3—silorane-based resin composite/self-etch adhesive (SBC-SE) (Filtek LS Low Shrink Posterior Restorative System/LS self-etch adhesive). All restorative procedures were done according to the manufacturer’s specifications (Table 1). The resin composites were placed in four oblique increments, and light-cured for 40 seconds with an LED curing-light unit (SmartLite™ PS, DENTSPLY, www.dentsply.com) with light intensity of 950 mW/cm². Restorations were immediately finished with a fine-grit diamond finishing bur #133 F (H133-010, 8-Fluted,

TABLE 1

Experimental Groups with Application Mode, According to Manufacturer’s (3M ESPE) Instructions

Group	Material	Composition	Adhesive	Procedure
1 (DBC-ER)	Filtek™ Supreme Ultra (Lot: N142477)	Resin: bis-GMA, UDMA, TEGDMA, bis-EMA, PEGDMA Filler: 72.5 wt. %; 55.6 vol. % zirconia/silica	Adper™ Single Bond Plus (Lot: N162108) Total-Etch	Etch enamel with Scotchbond™ etchant (35 wt. % phosphoric acid) for 15 seconds; rinse with water for 10 secs; blow dry and immediately apply two coats of adhesive using applicator; air dry for 5 secs to evaporate solvents; photocure for 10 secs; resin placed in <2 mm increments and cured for 20 secs; finished using #133F bur and polishing discs
2 (DBC-SE)	Filtek™ Supreme Ultra (Lot: N142477)	Resin: bis-GMA, UDMA, TEGDMA, bis-EMA, PEGDMA Filler: 72.5 wt. %; 55.6 vol. % zirconia/silica	Adper™ Easy Bond (Lot: N130974) Self-Etch	Apply self-etch adhesive using an applicator for 20 secs, and then air-thin for 5 secs; photocure for 10 secs; resin placed in <2 mm increments and cured for 20 secs; finished using #133F bur and polishing discs
3 (SBC-SE)	Filtek™ LS (Lot: N143481)	Resin: polysilorane Filler: 53 vol. %; 73 wt. % quartz	Silorane system adhesive (2-step, self-etch)	Apply self-etch primer for 15 secs; air dry and photocure for 10 seconds; apply bonding agent, air spread, and photocure for 10 secs; apply composite in 2 mm increments and photocure for 40 secs; finished using #133F bur and polishing discs

NTI, www.nti.de) under water cooling and polished with the last two finest grits of a series of flexible discs (Sof-Lex™ XT Pop-On, 3M ESPE).

MICROLEAKAGE EVALUATION

Teeth were stored in distilled water at 37°C for 7 days and then were thermocycled for 5000 cycles in separate distilled water baths of 5°C and 55°C ± 2°C with a dwell time of 30 seconds. The apex of each tooth was then sealed with resin composite (Filtek Supreme Ultra/Adper Easy Bond Self-Etch Adhesive) and the roots were embedded in epoxy resin. The entire surface of each tooth was then coated with two layers of nail varnish, to within 1 mm of the restoration. The teeth were then immersed in 0.2% methylene blue dye for 24 hours at room temperature, removed, and thoroughly rinsed with running tap water. The samples were then sectioned longitudinally in an occlusal-gingival direction using a low-speed diamond saw with water cooling (IsoMet, Buehler Ltd., www.buehler.com). Each section was examined at 10x magnification under a Meiji (Meiji Techno Co., Ltd., www.meijitechno.com) binocular microscope. Two independent observers blindly assessed the microleakage. The occlusal and gingival margins were scored based on depth of dye penetration, as described in Table 2.

MICROTENSILE BOND STRENGTH (μ TBS)

Flat dentin surfaces were created on mid-coronal dentin using a low-speed diamond saw (IsoMet, Buehler Ltd.). To create a standard smear layer, the exposed dentin was polished with 600-grit silicon carbide sandpaper for 60 seconds. A 4-mm-thick resin composite block was placed over the exposed dentin of each tooth following the treatment proposed for the experimental groups (Table 1). Resin composite was placed in two increments of 2 mm and light-cured for 40 seconds (SmartLite PS) with a light intensity of 950 mW/cm². After storage in distilled water at 37°C for 24 hours, each tooth was attached to an acrylic block for sectioning using a low-speed diamond saw (IsoMet) under continuous water irrigation to produce serial sticks with a cross-sectional area of approximately 0.9 mm². The first series of cuts through the bonded interface was in a mesio-distal direction made parallel to the longitudinal surface of the tooth. The second series of cuts was made perpendicular to the previous cuts in a bucco-lingual direction. The last cut was oriented transversally to the longitudinal surface in order to liberate the sticks. The specimens were measured using a digital caliper (Mitutoyo digital calipers, Mitutoyo America, www.mitutoyo.com) to calculate the bonded area in square millimeters and discard the imperfect specimens, in order to obtain approximately 30 specimens per group with similar dimensions. The specimens were tested individually by attaching them in a microtensile device using cyanoacrylate glue (Zapit, DVA, www.dentalventures.com). The specimens were submitted to a tensile force in an Instron® machine (Instron, www.instron.us) with a crosshead-speed of 0.5 mm/min until

TABLE 2

Dye Penetration Score Criteria

Score	Criteria
0	No dye penetration
1	Dye penetration up to one-half the cavity wall length
2	Dye penetration up to the full length of the cavity wall, not including the axial wall
3	Dye penetration to the full length of the cavity wall, including the axial wall

fracture. The force (N) and the bonding surface area (mm²) were recorded to calculate the μ TBS in megapascals (MPa).

STATISTICAL ANALYSIS

To compare the quantity of leakage at the enamel and dentin margins among the experimental groups, Kruskal-Wallis tests were used. While among-group differences were statistically significant, pairwise comparisons were made using Wilcoxon two-sample tests with a Bonferroni correction for multiple comparisons. Statistical comparisons were considered to be statistically significant at the 0.05 level. SAS 9.2 (SAS Institute, www.sas.com) was used to analyze the data. Samples subjected to the microtensile test were analyzed using ANOVA and Tukey's B rank order test ($\alpha = 0.05$).

SCANNING ELECTRON MICROSCOPY

Representative samples were prepared and restored as above to illustrate the hybrid layer and bonding interface of the three different groups. Teeth with Class V restorations were sectioned occluso-gingivally using a low-speed diamond saw (IsoMet). Samples were polished sequentially using 600-, 1000-, and 4000-grit silicon carbide paper, then etched with 5% maleic acid for 20 seconds, rinsed thoroughly with water, and dehydrated using increasing concentrations of ethanol (30% 30 min; 50% 30 min; 95% 2 hours; 100% 1 day). Subsequently, each sample was critical-point dried, mounted on a 12-mm aluminum stub using a cyanoacrylate adhesive, and sputter-coated in platinum to 8-nm thicknesses. The adhesive interface was analyzed by scanning electron microscopy at 10 kV accelerating voltage and up to 5000x magnification (S-2500 Hitachi Scanning Electron Microscope, Hitachi High Technologies America, Inc., www.hitachi-hita.com).

Results

MICROLEAKAGE

Table 3 shows the distribution of microleakage scores at the enamel and dentin margins. The Kruskal-Wallis test revealed

TABLE 3

Microleakage Score of Experimental Groups According to Margin Location

Group/ Score	Enamel (above cementoenamel junction)				Dentin (under cementoenamel junction)			
	0	1	2	3	0	1	2	3
1 (DBC-ER)	2	0	3	7	1	0	0	11
2 (DBC-SE)	0	5	2	5	2	4	1	5
3 (SBC-SE)	1	5	4	2	5	3	1	3

TABLE 4

Mean of Microtensile Bond Strength Values (MPa) and Standard Deviations for the Three Experimental Groups

Group	Microtensile Bond Strength (MPa)
1 (DBC-ER)	22.63 (± 5.60) ^a
2 (DBC-SE)	14.79 (± 3.94) ^b
3 (SBC-SE)	12.06 (± 5.25) ^b

Mean MPa with the same letters indicate groups that were not statistically different ($P > 0.05$)

no significant difference among the experimental groups at the enamel margin ($P = 0.191$). At the dentin margins, there was a statistically significant difference between the groups ($P = 0.008$). Pairwise comparison of groups at the dentin margins showed significant difference after a Bonferroni correction between the silorane/self-etch restorative system and the dimethacrylate/etch-and-rinse restorative system with the SBC-SE exhibiting less leakage than the DBC-ER ($P = 0.008$). The differences between SBC-SE and DBC-SE, and between DBC-ER and DBC-SE were not statistically significant.

MICROTENSILE BOND STRENGTH

One-way ANOVA showed significant differences for the experimental groups ($P < 0.05$). Mean microtensile bond strengths with standard deviations are given in Table 4. Tukey’s B rank order test showed that Group 1 (DBC-ER) presented the highest μ TBS, which was significantly different from both the DBC-SE and SBC-SE. Group 2 (DBC-SE) and Group 3 (SBC-SE) were not significantly different from each other ($P > 0.05$).

SCANNING ELECTRON MICROSCOPE

Illustrative scanning electron micrographs from each experimental group can be visualized in Figure 1 through

Figure 3. While the measurement of the hybrid layer and the quantification of resin tags was beyond the scope of this study, formation of resin tags can be visualized into the dentinal tubules on the etch-and-rinse and self-etch adhesive systems.

Discussion

MICROLEAKAGE

The first null hypothesis tested in the present study should be partially rejected, because the silorane resin composite with its dedicated self-etch adhesive system (SBC-SE) presented significantly less microleakage compared to dimethacrylate-based composites with an etch-and-rinse adhesive system (DBC-ER) at the dentin margin. This finding is in agreement with other studies.¹⁴⁻¹⁶ Although these studies have used slightly different comparative composite/adhesive systems, aging methods, dye infiltration protocols, and cavity preparations, SBC showed results comparable to or significantly better than conventional DBC in terms of microleakage. Nevertheless, some of the studies cited above failed to demonstrate a comparison with a self-etch adhesive system. In the present study, a DBC with a self-etch adhesive system was included as a comparison with SBC, which uses its dedicated self-etch adhesive system. While SBC performed better than the DBC when combined with an etch-and-rinse adhesive system at the dentin margin, there was no significant difference between these two restorative systems (SBC and DBC) when the self-etch versions were employed (enamel and dentin margins). These results agree with Ernst et al,¹⁵ who observed similar results of microleakage between SBC and DBC with self-etch adhesive systems at the cementum margins. It is important to mention that in the latter study, Class V cavity preparations with dimensions and location similar to those prepared in the present study were employed. Contrary to these findings, Yamazaki et al¹⁶ reported similar degrees of microleakage for SBC-SE and DBC with self-etch and etch-and-rinse adhesive systems. However, in their study, Class I cavity preparations were employed, excluding the microleakage evaluation below the CEJ. The evaluation of microleakage below the CEJ is relevant, because dentin is a less favorable substrate than enamel for adhesive bonding.^{14,15}

It is worth mentioning that when comparing microleakage results, some factors such as storage time and hydrophilicity of the adhesive systems can interfere with the results. According to Ernst et al,¹⁵ the comparison of marginal integrity among different composite/adhesive systems should ideally be performed with adhesives with the same level of hydrophilicity, since it could have an influence on dye absorption. Silorane resin composites are considered more hydrophobic than dimethacrylate resin composite systems, due to the silorane’s added silane moiety.⁵ Nevertheless, the two-step self-etch dedicated silorane adhesive consists of a hydrophilic methacrylate-based primer, which should be applied and light-cured separately prior to the application of the adhesive layer, which is hydrophobic and also methacrylate-based (manufacturer

data). It has yet to be determined whether silorane composite's hydrophobic barrier can actually influence dye absorption/microleakage, since hydrophilic methacrylate monomers, such as HEMA (hydroxy ethyl methacrylate), are still components of the silorane dedicated primer formulation, in order to ensure bonding with the hydrated dentin.¹⁷ The high hydrophilicity of the silorane primer was previously reported, whereas the silorane bonding layer was considered quite hydrophobic, with the presence of hydrophobic bifunctional monomers to match the hydrophobic silorane resin.^{17,18}

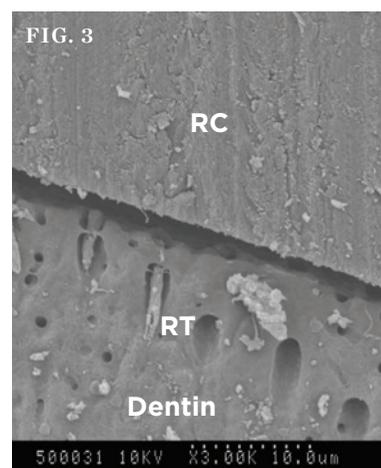
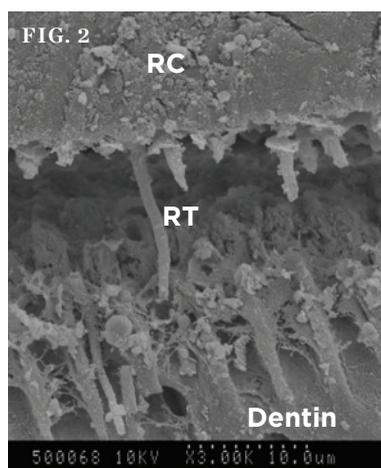
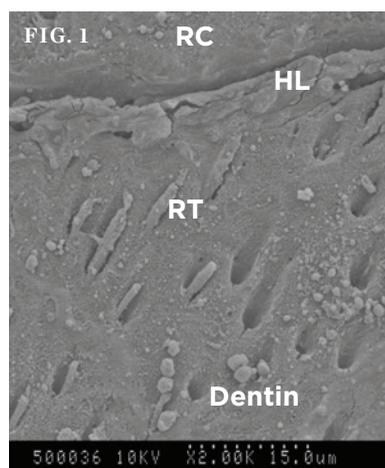
MICROTENSILE BOND STRENGTH

The second null hypothesis, which stated no significant difference in μ TBS among SBC and DSC restorative materials using self-etch and etch-and-rinse adhesive systems was rejected. The etch-and-rinse adhesive system combined with the dimethacrylate-based resin composite (DBC-ER) demonstrated the highest μ TBS values, which were significantly higher than the values obtained by the self-etch adhesive systems tested, including both DBC-SE and SBC-SE combinations. These results agree with other studies in which the μ TBS of etch-and-rinse adhesive systems outperformed the self-etch systems.^{11,12,19} Krajangta and Srisawasdi²⁰ verified that the silorane adhesive system used in combination with the silorane resin composite resulted in lower bond strength when compared to dimethacrylate-based resin composite with an etch-and-rinse adhesive, in spite of the low-shrinkage characteristic of the silorane restorative material. When self-etch versions from both silorane- and dimethacrylate-based restorative systems were compared, a similar performance in terms of microleakage and μ TBS was observed, showing no statistically significant difference between them. Boushell et al⁹ also reported similar

bond strength values for silorane- and dimethacrylate-based composite when self-etch adhesive systems were tested. The comparable results obtained by the self-etch adhesive systems in this study could be explained by the similar composition and “modus operandi” of these self-etch products. Although Filtek LS Silorane composition is based on a combination of siloxanes and oxiranes blocks, the silorane LS Adhesive System is based on the radical polymerization of methacrylates (technical information from 3M). Both the LS Adhesive System and Easy Bond are considered to be “ultra-mild” self-etch adhesives due to their high pH.^{21,22} The weak acids from the “ultra-mild” self-primers of these simplified adhesives do not remove the smear layer, but interact with it only up to hundredths of a nanometer.^{11,18} One possible advantage of these mild and ultra mild self-etch adhesive systems may be the preservation of the hydroxyapatite zone, leaving calcium available for possible additional chemical interaction with some specific adhesive functional monomers, which has been considered beneficial for the stability and longevity of the adhesive bonds.^{11,18}

In order to illustrate the interaction of the adhesive systems with the dentin complex, SEM micrographs were taken from each adhesive system (Figure 1 through Figure 3). The hybrid layer is considered a complex structure resulting from the chemistry of different adhesive systems and their application procedures.¹⁷ According to Ding et al,¹⁹ the presence of a thicker hybrid layer with numerous resin tags had a significant positive correlation with the μ TBS results obtained in their study. On the other hand, other studies have demonstrated that neither the thickness of the hybrid layer nor the length of the resin tags seem to have any influence on the bond strength of the adhesive systems.^{23,24} Although the quantification of resin tags was beyond the scope of

FIGURE 1 THROUGH FIGURE 3. Cross sections of evaluated restorative materials at the resin/dentin interfaces displaying resin composite (RC), resin tag (RT), hybrid layer (HL), and dentin. Group 1 (DBC-ER) under 2000x magnification (Fig 1); Group 2 (DBC-SE) under 3000x magnification (Fig 2); Group 3 (SBC-SE) under 3000x magnification (Fig 3).



this study, it is pertinent to report the presence of a hybrid layer with resin tags when both etch-and-rinse and self-etch systems were employed.

Conclusions

Within the limitation of this laboratory study, the first null hypothesis was partially rejected. The novel silorane-based resin composite and its dedicated adhesive system (SBC-SE) presented significantly less microleakage when compared to a dimethacrylate-based resin composite combined with an etch-and-rinse adhesive system (DBC-ER) at the dentin margin.

The second null hypothesis was rejected. A dimethacrylate-based resin composite combined with an etch-and-rinse adhesive system exhibited the highest μ TBS, which was significantly higher than the self-etch adhesive systems tested, including both dimethacrylate-based and silorane-based resin composite restorative systems.

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