

Durability of silorane repair with silorane and methacrylate resin

Durabilidade do reparo de resina composta à base de silorano realizado com a mesma ou com resina à base de metacrilato

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ABSTRACT

Objective: The durability of bonded interface between silorane (SIL) and methacrylate-based (MET) composite resins wasn't investigated yet. The purpose was to evaluate the repair strength (RS) and nanoleakage of the SIL repair using silorane- or methacrylate-based materials immediately [IM] or after 6 months of water storage [6M]. **Material & Methods:** fifteen resin blocks (Filtek P90, 3MESPE) were polished, sandblasted with 50- μ m aluminum oxide sandblasting and cleaned with 37% phosphoric acid. Then the specimens were divided into three groups: SIL-SIL (adhesive and composite resin); SIL-MET (silorane adhesive and methacrylate based composite) and MET-MET (methacrylate-based adhesive and composite resin) were used for the repair. Composite-composite bonded sticks (0.9 mm²) were sectioned and divided to be tested in tension (1.0 mm/min) IM or 6M. Two sticks from each tooth, at each time, were immersed in a 50% silver nitrate solution and evaluated by SEM. Data were analyzed by two-way repeated measures ANOVA and Tukey's tests. **Results:** The RS of the SIL was not dependent upon the adhesive and composite used for the repair. A significant reduction of the RS was observed for all groups after 6M. **Conclusion:** Both materials can be used for the repair of the silorane composite, despite degradation occur after 6M.

KEYWORDS

Composite resins; Methacrylates; Silorane resins.

RESUMO

Objetivo: A durabilidade da interface de união entre resinas à base de silorano (SIL) e à base de metacrilato (MET) ainda não foi estudada. O objetivo foi avaliar a resistência de união ao reparo de (RS) e nanoinfiltração da resina SIL com ela mesma ou com MET imediatamente (IM) ou após 6 meses de armazenamento em água (6M). **Material e Métodos:** quinze blocos de resina composta (Filtek P90, 3MESPE) foram confeccionados, polidos, asperizados com jato de óxido de alumínio 50- μ m e limpos com ácido fosfórico 37%. Em seguida, foram divididos em três grupos: SIL-SIL (adesivo e resina à base de silano); SIL-MET (adesivo à base de silano e resina à base de metacrilato) and MET-MET (adesivo e resina à base de metacrilato) para o reparo. Foram seccionados palitos de aproximadamente 0,9 mm², divididos entre IM e 6M e levados para máquina de microtração (1,0 mm/min). Dois palitos de cada bloco, de cada tempo, foram imersos em nitrato de prata 50% e avaliados em MEV. Os dados foram avaliados por ANOVA de dois fatores e teste de Tukey. **Resultados:** A RS da resina SIL não foi dependente da resina ou do adesivo utilizado para o reparo. Uma redução significativa da RS foi observada para todos os grupos após 6M. **Conclusão:** ambos os materiais podem ser usados para reparo de resinas SIL, apesar de a degradação ocorrer após 6M.

PALAVRAS-CHAVE

Resinas compostas; Metacrilatos; Resinas de Silorano.

INTRODUCTION

Tooth-colored composite materials have grown in popularity over the last few decades. Apart from the development of a minimal invasive preparation technique as well as improved adhesion to tooth structures these materials exhibit predictable long-term-stability with annual failure rates that are comparable for composites and amalgam in stress-bearing class I- and class II-cavities [1].

Methacrylate-based composites exhibit acceptable clinical performance, but several negative effects in resin-based composite restorations, like marginal discrepancies [2,3], marginal staining, white lines around the restoration, deformation of the cusps [4], debonding [5], microleakage [6], recurrent caries, postoperative sensitivity or pain [7] are frequently related to polymerization shrinkage stress.

Several approaches have been proposed to reduce shrinkage stress such as controlling the cavity configuration (C-factor) [8], modulating the light intensity using different polymerization techniques [9], using different cavity filling methods [10], and applying stress-absorbing intermediate layers [11]. All these approaches are intended to be used with the methacrylate-based resins. In the last few years, a new restorative material was developed in order to reduce these disadvantages.

The silorane-based composite resin is based on the innovative monomer system obtained from the reaction of the oxirane and siloxane molecules [12,13], which exhibit similar or better mechanical and physical properties than conventional methacrylate-based composite resins: low polymerization shrinkage (less than 1.5%) [12,14], lower polymerization shrinkage stress [15], flexural strength, dynamic and static moduli of elasticity, hardness [16,17], wear resistance [18,19], biocompatibility (non-mutagenic and non-clastogenic) [20] and better overall color stability over time [21].

Regardless of the monomer system employed on the composite resin, these restorative materials still deteriorate and degrade over time, as do the majority of dental materials. Actually, consistent with the philosophy of "minimum intervention" operative dentistry, failures of the restoration have been repaired instead of replaced. This is cost-effective, requires less clinical time, allows preservation of tooth dental structure, and minimizes pulp damage [22,23]. Thus, defective restorations first should be first evaluated for repair possibilities, rather than routinely replaced [24]. Clinical studies involving composite resin repairs have shown that when properly planned, the repairs may increase the clinical longevity of restorations, representing a conservative choice for treatment of restorations [25-27].

However, it is not possible to distinguish between silorane and methacrylate-based composite resins with the clinician's tools. Therefore, the knowledge of the bonding between these two resins is important, mainly in the case of the former needing repair. So far, few studies have attempted to evaluate the repair bond strength between these two kinds of resin [28-30], and the ones available have not focused on the durability of such bonded interface. Therefore, the aim of this study was to investigate the immediate and 6-month repair bond strength between silorane- and a methacrylate-based composites as well as the nanoleakage in the bonded interface.

MATERIAL & METHODS

Fifteen resin composite blocks were made by layering 2 mm thick increments of a silorane-based composite resin (shade A3, P90 [Filtek Silorane as available outside Brazil], 3MESPE, St. Paul, MN, USA) in an addition silicone mold (4 mm high and with a diameter of 12 mm). Each increment was condensed with a clean plastic filling instrument to avoid contamination and light-cured for 40 s (Optilux®, SDS Kerr, Middleton, Wisconsin, USA) with an output of 600 mW/cm². The last increment was covered

and compressed with a glass microscope slide in order to obtain a flat surface. Each specimen was removed from the mold and the surfaces of the composite blocks were manually polished with the DiamondPro (FGM Produtos Odontológicos, Joinville, SC, Brazil). Each of the four disks was applied manually for 10 s in a single direction on the surface with constant and intermittent pressure. This procedure was extensively studied in a pilot study in order to standardize the procedure. After this, specimens were stored in a dark vial with water at 37 °C for one week.

All surfaces were sandblasted with 50 μm aluminum oxide powder for 10 s at a working distance of 5 mm at a pressure of 5.5 Pascals (Pa) with an intraoral sandblaster (Microetcher II, Danville Engineering Inc, San Ramon, CA, USA). For purposes of cleaning, a 37% phosphoric acid etchant (Condac, FGM Produtos Odontológicos) was applied for 30 s. After water-rinsing (30 s) and air-drying (10 s), specimens were randomly assigned to three groups according to the repair materials to be used.

In the **SIL-SIL group** (control), the Filtek Silorane System Adhesive (3M ESPE) was applied according to the manufacturer's purpose (Table 1). After adhesive application, two 2-mm increments of the silorane composite (P90, shade A2, 3M ESPE) were placed over the treated surfaces and each increment was light-cured with the same light-curing device for 40 s. In the **SIL-MET group**, the silorane adhesive was applied and instead of using the silorane resin, a methacrylate-based composite (shade A1, Opallis, FGM Produtos Odontológicos) was placed in 2-mm increments as previously reported. In the **MET-MET group**, the methacrylate adhesive (Ambar, FGM Produtos Odontológicos) was applied according to the manufacturer's instructions (Table 1) and the methacrylate-based Opallis composite resin (shade A1, FGM Produtos Odontológicos) was placed as described for the other groups.

Bonded composite-composite samples were longitudinally sectioned with a slow-speed diamond saw (Isomet; Buehler, Lake Bluff, IL,

USA) under water cooling in both "x" and "y" directions across the bonded interface to obtain bonded sticks with a cross-sectional area of approximately 0.9 mm². The number of composite-composite sticks that failed during specimen preparation was recorded. Specimens were then divided to be tested either immediately [IM] or after 6 months [6M] of storage in distilled water at 37 °C as was usually done in interfacial degradation studies of resin-dentin bonds. During the 6-month storage period, water was not regularly refreshed, but in case we detected water evaporation from the vial, water was added to keep a constant water volume.

The area of each stick was measured with a digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan) for calculation of the CR strength. They were attached to the universal testing machine (Instron, Instron Corp., Canton, Mass., USA) with cyanoacrylate resin (Super Bonder gel, Loctite, São Paulo, SP, Brazil) and subjected to a tensile force (1.0 mm/min) for evaluation of the microtensile repair strength. The failure modes were evaluated at 400X (HVM-2, Shimadzu, Tokyo, Japan) and classified as: 1. CSi, for failures exclusively within the silorane composite; 2. CMet, for failures exclusively within the methacrylate resin; 3. A, for adhesive failures and 4. PF, for premature failures.

Nanoleakage (NL) under Scanning Electron Microscopy (SEM)

Two bonded sticks from each composite block at each storage period were not tested under tension, but were kept to use for NL. They were coated with two layers of nail varnish applied up to within 1 mm of the bonded interfaces. After re-hydration in distilled water for 10 min, they were immersed in an aqueous solution of 50 wt% of silver nitrate and left in darkness for 24 h, rinsed thoroughly in distilled water and immersed in a photo-developing solution for 8 h under a fluorescent light. Specimens were wet polished using SiC paper with decreasing grit (1000, 1200, 1500, 2000, 2400) and 1 and 1/4 μm diamond paste (Buehler

Ltd, Lake Bluff, IL, USA) using a polish cloth. They were ultrasonically cleaned, air dried, mounted on aluminum stubs and sputtered with gold to be evaluated by SEM (JSM 6060, JEOL, Tokyo, Japan) in the backscattered mode with an accelerating voltage of 20 KV. Three pictures were taken of each specimen by a technician who was blinded to the experimental conditions under evaluation. The images were only qualitatively analyzed.

Statistical Analysis

The microtensile repair strength of all sticks with adhesive failure from the same resin block was averaged for statistical purposes. The specimens with cohesive and premature failures were not included into the tooth mean.

Before submitting the data to the appropriate statistical analysis, the Kolmogorov–Smirnov test was used to assess whether the data followed a normal distribution and the Barlett's test was used for assessment of the equality of variances. After observing the normality of the data distribution and the equality of the variances, the μ TBS (MPa) were subjected to a two-way repeated measures analysis of variance (Adhesive vs. Application mode vs. Storage time) and the hydraulic conductance (%) to a two-way analysis of variance ($\alpha = 0.05$). The Tukey's test was used for pair-wise comparisons ($\alpha = 0.05$). The data from the fracture pattern was analyzed by the chi-square test ($\alpha = 0.05$).

Table 1 - Materials, manufacture, batch number, composition and mode of application

| Materials (manufacture / batch number) | Composition | Mode of application |
|--|---|---|
| Filtek Silorane (3M ESPE/ 1208700531) | Silane treated quartz 60–70%, Yttrium trifluoride 5–15%, Bis-3,4-epoxycyclohexylethyl-phenyl-methylsilane 5–15%, 3,4-Epoxy-cyclohexylcyclo polymethylsiloxane 5–15% | (1) Individual increments with incremental technique should not exceed 2.5 mm; (2) Light cure each increment for 40 s (halogen 500 - 1400 mW/cm ²). |
| Filtek Silorane Adhesive System (3M ESPE/ 1134700248) | 70–80% Substituted dimethacrylate, 5–10% Silane treated silica, 5–10% TEGDMA, <5%, Phosphoric acid-methacryloxy-hexylesters, <3%, DL-camphor-quinone, <3% 1,6-hexanediol dimethacrylate | (1) Shake bottle briefly before dosing; (2) Place one drop of SSA-Bond in the applicator; (3) Use a gentle stream of air until SSA-Bond is spread to an even film and does not move any longer; (4) Cure SSA-Bond for 10 s. |
| Opallis (FGM/ 011210) | Bis-GMA monomers, Bis-EMA, TEGDMA, UDMA, camphorquinone, co-initiator, silanized barium-aluminum silicate glass (particle size of 0.5 μ m, 79.8 wt%), pigments and silica | (1) Individual increments with incremental technique should not exceed 2.0 mm; (2) Light cure each increment for 40 s. |
| Ambar (FGM/ 100511) | MDP; UDMA, 2-hydroxyethyl methacrylate, and other hydrophilic and acid methacrylate monomers, ethanol, silanated silica, photo-initiators, co-initiators, and stabilizers | (1) Place one drop of Ambar in the applicator; (2) The first coat should be vigorously agitated for 10 s; (3) Repeat the steps 1 and 2; (4) Gently air dry for 10 s; (5) Light-cure for 10 s using a light curing unit with minimum light intensity of 400 mW/cm ² . |

Abbreviations: Bis-GMA: bisphenol A diglycidyl methacrylate; Bis-EMA: bisphenol A diglycidyl methacrylate ethoxylated; TEGDMA: triethylene glycol dimethacrylate, UDMA: urethane dimethacrylate; HEMA: hydroxyethyl metacrylate; MDP: 10-methacryloxydecyl dihydrogen phosphate.

RESULTS

Approximately 30 to 35 composite-sticks were obtained per resin block, half were tested immediately and the other half were tested after 6 months of water storage. As five composite blocks were prepared for group, a total of approximately 65 to 80 resin-dentin sticks were tested per experimental condition.

The means and standard deviations of the repair bond strength values (MPa) are shown in Table 2. Only the main factor storage period was statistically different ($p = 0.02$). The groups observed at the immediate time showed higher repair bond strength values than those observed after 6 months of water storage.

Table 2 - Means and standard deviations of the repair bond strength (MPa) for the different experimental groups

| GROUPS | STORAGE TIME | |
|---------------------------|--------------|--------------|
| | IMMEDIATE | 6 MONTHS |
| SIL-SIL | 38.5 ± 3.9 | 28.4 ± 4.9 |
| SIL-MET | 35.0 ± 3.7 | 34.3 ± 5.4 |
| MET-MET | 38.9 ± 10.3 | 34.5 ± 4.4 |
| Main factor storage time* | 37.4 ± 6.5 A | 32.6 ± 5.2 B |

* Means identified with different uppercase letters are statistically different.

The fracture mode of all groups is shown in Table 3. Adhesive failure was more frequently observed than cohesive and premature failure for all groups at the immediate and 6-month periods

Table 3 - Fracture pattern distribution [number (Percentage)] according to the experimental groups

| GROUPS | STORAGE PERIOD | | | | | | | |
|---------|----------------|-------------|---------------|---------------|-------------|------|---------------|-------------|
| | IMMEDIATE | | | | 6-MONTH | | | |
| | CSi | CMet | A | PF | CSi | CMet | A | PF |
| SIL-SIL | 1 (1.1%) | - | 58 (63.7%) | 32 (35.2%) | 3 (4.7%) | - | 61 (95.3%) | - |
| SIL-MET | - | - | 58 (93.5%) | 4 (6.5%) | - | - | 53 (100%) | - |
| MET-MET | 2 (2.4%) | 3 (3.7%) | 71 (86.6%) | 6 (7.3%) | - | - | 71 (98.6%) | 1 (1.4%) |

CSi: cohesive failure within silorane; CMet: cohesive failure within the methacrylate resin; A: adhesive failure; PF: premature failures.

(chi-square test; $p < 0.0001$). However, one can see that in the immediate time, the bonded sticks from the SI-SI group showed the highest percentage of premature failures (35.2%) while the groups SI-MET and MET-MET showed a very low percentage of premature failures (6.5% and 7.3% respectively) (chi-square test; $p < 0.0001$).

A representative image of nanoleakage can be seen in Figure 1 (back-scattered SEM image of the composite-composite interfaces bonded in SIL-MET group). No silver nitrate deposition was seen for both adhesives in the immediate period and after 6 months of water storage.

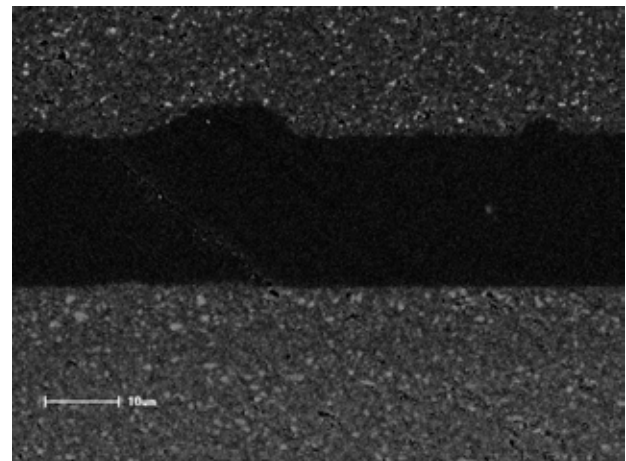


Figure 1 - Representative back-scattered SEM image of the composite-composite interfaces bonded in SIL-MET group. Note the absence of spotted silver nitrate deposits within the adhesive layer after six months of distilled water storage. All groups presented similar image.

DISCUSSION

Most authors repair the original composite with the same material [29]. However, as there are many restorative materials available in the market, the substrate for bonding repair is rarely known by the clinician and the composite used for the repair is likely different than the original one. This makes the clinical success difficult to predict.

So far, no earlier study attempted to evaluate the reparability of the silorane-based composite with the methacrylate-based composite resin Opallis (FGM Produtos Odontológicos). The repair microtensile bond strength values found in the study confirms previous literature findings that silorane-based composite can be repaired with different brands of methacrylate-based composite [31-34]. The compatibility between these two different composite resins was also recently reinforced in a 12-month clinical trial, which concluded that silorane-based composite exhibited a similar performance to conventional methacrylate-based composite when used to repair composite resin restorations [35].

Although there is an expected chemical affinity between the substrate of silorane and silorane-based adhesive and composite resin, this is not expected when the repair material is methacrylate-based. Based on this, the micromechanical coupling to the resin matrix is expected to be the cause of the obtained repair bond strength of the silorane with the methacrylate bonding and composite resin. Thus it seems of paramount importance to use clinical tools to enhance the micromechanical interlocking between the substrate and the repair material as clinicians will never be sure if they are from the same chemistry.

In the present study, the silorane-based composite was sandblasted with 50 μm aluminum oxide and etched with 37% phosphoric acid etchant prior to the application of the bonding resin. This technique was reported to produce higher repair bond strength for methacrylate-based composites [36-39]

and also for the silorane-based composite in earlier studies [34,40]. Similarly, other studies investigated the role of surface roughness and the brand of adhesive resin in the repair strength of silorane-based composite [32], concluding that these factors do not play an important role in the strength of the repair, since successful repair was obtained irrespective of the surface roughness and chemistry of the repair material used.

We did not use silane before adhesive application, because earlier studies showed that silane application did not in any way enhance on the repair strength of methacrylate-methacrylate [41,42] and silorane-methacrylate-based composites [32,33]. However, this still deserves further investigations since some authors reported that if the original composite is silorane-based, then using the silane-based adhesive may be the best repair option [28,29].

Although the repair strength of the silorane-based adhesive was not affected by the chemistry of the bonding resin and composite resin, the repair strength reduced significantly after 6 months of water storage. This seems to be related to the hydrophilicity of the bonding resin, either based on silorane or methacrylate chemistry, used for the repair of the silorane-based composite. Bonding resins are expected to be hydrophilic to interact with the moist dentin substrate. Thus, they are highly prone to water sorption [43,44].

The absorbed water causes swelling of the polymer structure at the adhesive interface and leads to plasticization and softening of the resin structure [45]. Then, unreacted monomers and oligomers trapped in the polymer network are released to their surroundings, which create micro-voids that may be stained with silver nitrate. Although the present study did not detect silver nitrate deposition in any of the adhesive interfaces, this may be due to the short-term period of water immersion. After aging these substances for a prolonged period (2 years), the authors of a recent study detected small areas of silver nitrate deposition at the interface in the substrate side, which represents signs of degradation [32].

The high percentage of premature failures in the SI-SI group was quite unexpected since all the materials in this group were from the same chemistry. A silorane-based composite presents a lower degree of conversion than a methacrylate-based composite [46-48] which would theoretically favor bonding of the repair material with the same chemistry. However this does not seem to occur with silorane-based materials. The relatively lower DC values are likely related to tetrafunctionality of silorane molecules. In multifunctional monomers, the reactivity of a macro-radical decreases greatly when one of the molecules reacts, because as the molecule is trapped in the network under formation, the mobility of other functionalities decreases [49]. Thus, we may hypothesize that the bonding of silorane-based repair material on a cured and aged silorane substrate is more challenging than bonding a methacrylate-based repair material on a cured and aged methacrylate substrate. However, further studies should be conducted to elucidate this issue.

CONCLUSION

The silorane-based composite can be repaired with the same material or with a dimethacrylate-based composite, but similar to other bonding materials, the adhesive layer degraded after 6 months of water storage. Although no nanoleakage was observed in any of the adhesive interfaces, long-term water immersion periods should be tested to confirm this finding.

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