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ORIGINAL ARTICLE

The effect of surface treatment and bonding procedures on the bond strength of silorane composite repairs

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Abstract

Objective. The aim of this study was to evaluate the effect of surface treatments and adhesive protocols on the microtensile bond strength of a low-shrinkage composite repair. ***Materials and methods.*** Ninety-six blocks of composite resin Filtek LS were prepared using a half-hourglass-shaped silicone matrix. The specimens were storage for 24 h in distilled water and were randomly divided into the experimental (6) and negative control (2) groups ($n = 12$) according to the surface treatment (diamond bur and aluminum oxide sandblasting) and adhesive protocol (none; Filtek LS adhesive; phosphoric acid + Filtek LS adhesive; and phosphoric acid + silane + Filtek LS adhesive). After the adhesive procedure, the specimens were fixed in an hourglass-shaped silicone matrix and the other half of the specimen was restored. Hourglass shaped specimens ($n = 12$) were used as positive control (cohesive strength of the resin). The microtensile bonding test was performed at a crosshead speed of 0.5 mm/min. The data were analyzed using ANOVA, Tukey's and Dunnett's tests ($\alpha = 0.05$). ***Results.*** The bond strength values were similar for all experimental groups, except the groups without adhesive application. None of the experimental groups presented results similar to the positive control group. ***Conclusions.*** The repair of silorane restorations is viable; nevertheless, the different bonding procedures tested were incapable to produce bond strengths similar to the cohesive strength of the material.

Key Words: *Composite resin, adhesive system, microtensile, silane*

Introduction

Composite resins are widely used for direct restorative treatment; however, failures may occur and lead to unsuccessful clinical outcomes, such as fractures, marginal staining, color change, anatomical deficiencies, recurrent caries and dentin sensitivity/pain [1].

For failures that don't compromise the structural integrity or adhesive interface, some studies shows that the repair is a viable alternative, since the bond strength obtained through this procedure may result in a similar cohesive strength of the composite to that of the material used in the restorative treatment [2–4]. The great advantage of the repair is maintenance of the sound dental tissue surrounding the restoration, which are removed in the entirely substitutions, even unintentionally, due to the close contact of the restorative interface and healthy tissue. Regarding the clinical procedures, several studies have shown that the association of surface treatment and adhesive systems can significantly increases the bond strength between pre-existing resin and the composite repair [2–8].

Silorane-based composites are low-shrinkage materials, which contain a monomer system with a cationic ring-opening polymerization [9]. This process results in low polymerization shrinkage, which is a relevant factor for restorative treatment, since high shrinkage may be responsible for gap formation in the adhesive interface [10] and increased microleakage in the restorations [11,12].

The success of a repair depends on obtaining a suitable adhesive interface between the new and

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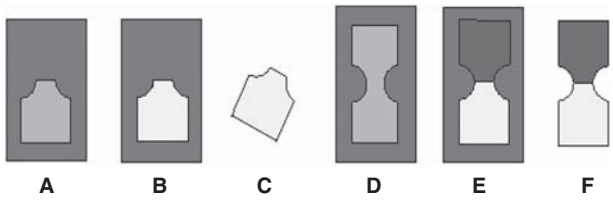

Figure 1. Scheme representing different stages of specimen preparation: half-hourglass-shaped silicone matrix (A); preparation of the specimen prior to restoration (B) and specimen representing the restoration (3 mm base, 1 mm thick and constricted area of 1 mm^2) (C). Hourglass silicone matrix for preparing the repair (D); repair prepared in silicone matrix (E) and, after removing this, with an interface showing an area of 1 mm^2 (F).

pre-existing composite resin. Viability analysis of repairs in restorations with silorane-matrix composites is important to obtain crucial information regarding the best way to perform the procedure obtaining the higher bond strength. Therefore, the aim of this study was to evaluate the effect of different surface treatments on the microtensile bond strength of a silorane-based composite repair. The first hypothesis tested was that the different surface treatments and adhesive protocols would not present differences in bond strength; the second hypothesis was that the bond strength of the repairs would be similar to the cohesive strength of the silorane composite.

Materials and methods

Specimens preparation

Ninety-six specimens of low-shrinkage composite resin (Filtek LS, 3M ESPE, St. Paul, MN), shade A2, were prepared using a half-hourglass-shaped silicone matrix [3,7] (Figure 1A, constricted area of 1 mm^2).

The silicone matrix was filled with composite resin and then covered with a Mylar strip and a microscope slide. To compress the material and prevent bubble formation, the glass slide was gently pressed. The composite was polymerized for 40 s, according to the manufacturer's recommendations, using a halogen light-curing unit (Optilux 501; Sybron Kerr, Danbury, CT) at 650 mW/cm^2, which was monitored by a radiometer (model 100; Demetron/Kerr, Danbury, CT). The specimens were removed from the matrix, and the area to be repaired was finished using abrasive discs (Soflex, 3M ESPE), to remove the residual monomers present in this surface. All specimens were stored for 24 h in distilled water at 37°C [2] and were randomly distributed into six experimental groups and two negative control groups (n = 12) according to the surface treatment and adhesive protocol used, as described in Table I.

As a positive control group, hourglass shaped specimens (n = 12) were prepared to evaluate the cohesive strength of the material.

Preparation and surface treatment of the repaired slabs

The roughening with a diamond bur was performed on the entire bonding surface of the specimens in groups 6, 7, 8 and 9 using a #3098 bur (regular grit, KG Sorensen, Barueri, SP, Brazil) using a high-speed handpiece and under constant water-cooling [2]. For the surface treatment with aluminum oxide sandblasting (groups 2, 3, 4 and 5), a sandblasting handpiece (Microetch Bioart, São Carlos, SP, Brazil) with 50 μm sized aluminum oxide particles was used, at a distance of 5 mm for 10 s [2]. Then the surface was washed with distilled water and dried for 15 s.

The different adhesive treatments were performed as follows:

- Filtek LS adhesive system (self-etching): primer application for 15 s with gentle air and light curing for 10 s. The Filtek LS bond was applied and light cured for 10 s.
- Phosphoric acid conditioning + Filtek LS adhesive system: the surface to be bonded was etched with 35% phosphoric acid (Scotchbond, 3M ESPE) for 30 s, washed for 15 s and dried. The primer was applied for 15 s and was followed by a gentle blast of air and light curing for 10 s. The Filtek LS bond was applied and light-cured for 10 s.

Table I. Experimental and control groups, according to the surface treatment and adhesive protocol.

Group	Surface treatment	Adhesive protocol
1	No treatment (positive control)	—
2	Aluminum oxide sandblasting (negative control)	—
3	Aluminum oxide sandblasting	Filtek LS adhesive system
4		Phosphoric acid + Filtek LS adhesive system
5		Phosphoric acid + Silane + Filtek LS adhesive system
6	Diamond bur (negative control)	—
7	Diamond bur	Filtek LS adhesive system
8		Phosphoric acid + Filtek LS adhesive system
9		Phosphoric acid + Silane + Filtek LS adhesive system

Table II. Bond strength means (MPa) and SD (standard deviation) of the silorane resin-based repairs, according to the surface treatment and adhesive protocol.

	Adhesive protocol			
Surface treatment	Without adhesive system	Filtek LS adhesive system	Phosphoric acid + Filtek LS adhesive system	Phosphoric acid + Silane + Filtek LS adhesive system
Aluminum oxide sandblasting	4.54 (2.59)Ba	21.35 (6.24)Aa	19.3 (6.69)Aa	21.78 (6.64)Aa
Diamond bur	3.72 (0.52)Ba	21.05 (6.45)Aa	18.92 (3.48)Aa	19.1 (6.28)Aa
Control	39.04 (6.08)*			

Capital letters compare the adhesive protocols and lower case letters compare the surface treatments (ANOVA two-way and Tukey's test; $\alpha = 0.05$). *Represents a statistical difference between the control and experimental groups (Dunnett's test; $\alpha = 0.05$).

- Silane application: the silane (Ceramic Primer, 3M ESPE) was applied and, after 1 min, the adhesive system was applied as described above.

To perform the specimen repair, a similar matrix was used; however, with an hourglass-shape (Figure 1D). After the surface treatment and/or adhesive protocol, the specimen was positioned in the matrix and the matrix was filled with the silorane composite resin. The restorative material used was a low-shrinkage composite resin, Filtek LS (3M ESPE, St. Paul), color C2, to allow the differentiation between the restoration and repair. The restorative technique was similar to that described above to prepare the restoration specimens.

Microtensile bonding test

The adhesive interface area was measured using a digital caliper to ensure the accuracy of the data. The specimens were fixed to a microtensile device coupled to a universal testing machine (EMIC, São José dos Pinhais, PR, Brazil), using a cyanoacrylate-based adhesive (Super Bonder gel - Loctite, São Paulo, SP, Brazil), in such a way that the interface area was perpendicular to the long axis of the tensile force. The test was performed at a crosshead speed of 0.5 mm/min. The microtensile bond strength values were obtained in Kgf (kilogram-force) and transformed into MPa (N/mm^2).

Failure mode analysis. After the microtensile bond-strength test, the fractured interfaces were evaluated by stereoscopic microscopy (45x, Meiji 2000, Meiji Techno, Saitama, Japan) to determine the failure mode of each restorative combination. The failure modes were classified into three types: (1) adhesive failure, (2) cohesive failure in the composite (corresponding to the restoration or repair) and (3) mixed failure (a combination of more than one type of fracture) [2].

Statistical analysis. The normal distribution of the obtained data was verified and the results were analyzed using 2-way analysis of variance (ANOVA) and Tukey's test ($\alpha = 0.05$). To compare all groups with the positive control group, Dunnett's test was used ($\alpha = 0.05$).

Results

The microtensile bond strength values are listed in Table II. The values were statistically similar for all experimental groups ($p = 0.83$), except the groups without the application of the adhesive system (negative controls). When compared to the positive control group, neither experimental group showed a bond strength similar to the cohesive strength of the composite resin ($p < 0.001$), which presented the highest values obtained in the present study.

The predominant failure mode found in all groups with adhesive application was the mixed failure mode. Adhesive failures were observed in all experimental groups in smaller amounts and no cohesive failures were found. The control group presented cohesive failures in all specimens. For the groups without adhesive application, the adhesive failure was observed in all specimens (Figure 2).

Discussion

The repair is an important characteristic of the composite resins, allowing one to re-establish some deficiencies in the restoration without complete removal of the adhesive material [2,13,14]. Therefore, the present study evaluated the low-shrinkage composite and its repair capacity after different surface treatments. Regardless, the surface treatment, the application of an adhesive layer was demonstrated, in the present study, to be crucial in improving the bond strength of the repair. Based on these results, the first hypothesis was rejected.

After specimen preparation, different treatment protocols were performed. According to the results, the surface treatments tested (diamond burs or sandblasting with aluminum oxide) presented similar bond strength values. Several studies have indicated that sandblasting with aluminum oxide produces

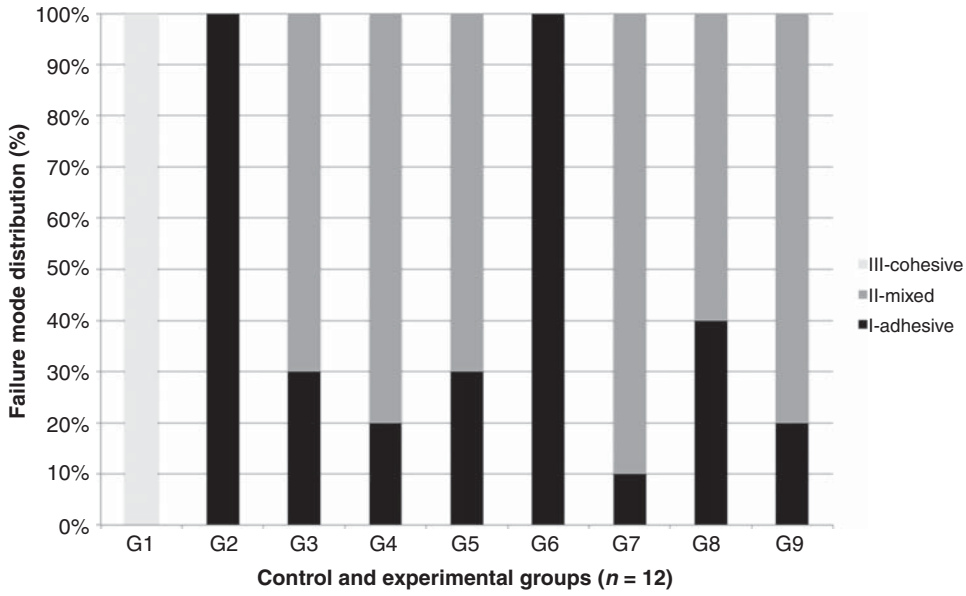

Figure 2. Failure mode distribution of the experimental and control groups.

more micro-retentive areas, increasing the surface area, improving the wetting and increasing the bond strength when an adhesive system is used [2,15–17]. Different to the sandblasting, the diamond bur produces 'micro' and 'macro' retentions that can favor the interlocking, using more viscous resins as intermediary agent or without application of an intermediary material [2]. However, the results of the present study demonstrate that, to perform the repair of the low-shrinkage used in the present study, both diamond bur and sandblasting with aluminum oxide can promote similar results of immediate bonding.

The use of phosphoric acid prior to adhesive application did not increase the bond strength of the repairs tested. This is due to the low efficiency of the acid used for surface conditioning [2,18]. Phosphoric acid is a weak acid and is incapable of causing changes in the composite resin surface, resulting in similar bond strengths to that of the groups where etching was not used. The inefficiency of acid with respect to improving the bond strength values of composite repairs was demonstrated in a previous study [2]. However, acid etching can be used to remove surface contamination by saliva or other agents in clinical situations.

Silane is an agent used in the ceramic cementation; it is a molecule with a functional group that promotes bonding between the silica present on the ceramic and the methacrylate of the resin cement or bonding agent [19,20]. However, in the present study, any influence of this agent was observed in the bond strength of the repairs, corroborating the finds of a previous study [21]. The use of a silane promoted results similar to the other groups, showing that the application of this agent cannot be necessary or, if performed, the agent can be used without jeopardizing the bond interface.

To evaluate the influence of the adhesive protocols in the bond strength of the silorane repair, the present study used two types of control groups. One analyzes the cohesive strength of the silorane composite (positive control), comparing the results with the bond strength of the different repair protocols. In this way, none of the experimental groups had similar bond strengths to the positive control group, and the second hypothesis must be rejected. A possible explanation for this result is due to the high cohesive strength of the composite resin tested. The presence of an interface, even with the application of an adhesive system, promotes a formation of a fragile point when a force is applied. Since the mechanical properties of the adhesives are reduced compared to the composite resins, the force needed for the rupture is inferior, promoting lower values of the bond strength, compared to the cohesive strength of the material. These data corroborate the results obtained in other previous studies using silorane and Bis-GMA based materials [2,7].

Experimental groups without application of an adhesive were used as negative control groups, to evaluate the influence of the bonding agents on the silorane repair. According to the results, it is confirmed that the adhesive layer is crucial for promoting an adequate silorane repair, regardless of the surface treatment, since the obtained results in the groups without bonding agents were lower than the groups with adhesive application.

The predominant failure mode observed in all groups with the applied adhesive was the mixed failure mode, except for the positive control group. Due to the presence of an adhesive layer, which promotes a suitable bonding between the two substrates, and the geometry of the specimens, probably the stress was concentrated in the adhesive interface, promoting

crack propagation in the adhesive, extending to the composite resin (repair or restoration), causing rupture in both substrates and promoting a mixed failure.

Some adhesive failures were observed, most probably due to the critical bonding procedure required by the silorane composite. The primer of the silorane self-etching adhesive is light-cured, unlike the conventional two-bottle self-etching adhesives. As a result, it should be speculated that small gaps between the primer and the bond agent can occur, causing premature failure of the interface, compromising the bonding.

For the groups without adhesive, the interfacial failure occurred in all specimens, due to the reduced bonding between the two substrates (the new and pre-existing resin). It should be noted that there was great number of premature failure (~ 40%) in the groups without adhesive application, highlighting the fragility of the interface, since any premature failure was observed in the groups using the bonding agent.

The repair of silorane restorations is possible and can be performed to re-establish deficient restorations without entirely removing those failed restorations, facilitating the procedure, making it faster and safer. However, the use of an adhesive layer is crucial and the bonding between the new and pre-existing resin is not similar to the cohesive strength of the silorane resin and these facts should be considered in the clinical decision.

Conclusions

According to the data obtained in the present study, it can be concluded that:

- The different surface treatments resulted in similar bond strength values.
- The adhesive layer is crucial to promote adequate bonding of the silorane composite repair.
- None of the experimental groups showed similar bond strengths when compared to the positive control group.

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