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Original Research Article

Repair on silorane-based composite

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Abstract

Introduction: Silorane-based composites have low polymerization shrinkage and good color stability. However, the effectiveness and the best surface treatment to carry out repairs to this type of restoration is unclear. **Objective:** To evaluate the effect of different types of repair made on a silorane-based composite. **Material and methods:** 80 disks of silorane-based composite were prepared (Filtek P90, 3M ESPE) and divided into eight groups (n = 10), according to the surface treatment being carried out before repairs of either the same silorane composite or a dimethacrylate material (Filtek Z350, 3M ESPE) were conducted. In two groups the immediate adhesion without repair (positive control) was evaluated. In other two groups repairs without any surface treatment (negative control) were evaluated. Surface treatments before the repair of the four remaining groups included the application of adhesive systems specific to silorane (Silorane System Adhesive, 3M ESPE) or to dimethacrylate (Adper Single Bond 2, 3M ESPE) and roughening followed by application of adhesive system. All groups were stored into distilled water at 37°C for 1 week prior to the microshear bond strength evaluation. **Results:** The group immediate adhesion silorane-dimethacrylate and group repair silorane-dimethacrylate without surface treatment showed lower microshear bond strength values and were statistically different from groups with surface treatment and immediate adhesion silorane-silorane ($p < 0.05$). **Conclusion:** Surface treatments with application of adhesive systems compatible with the repair material or roughening prior to the application of these adhesive systems are suitable for repairing silorane-based composites.

Introduction

Both the resin composites and the adhesive systems are very versatile and their use have favored the greatest advancements in aesthetic Dentistry leading to the preservation of tooth tissues and obtaining nearly invisible restorations by little wearing of the sound tooth structure. Thus, Dentistry has been currently experiencing the perspectives predicted for over one decade [19], by emphasizing restorative direct techniques and conservative preparations. However, the polymerization shrinkage of dimethacrylate-based composites has been considered as their major shortcoming resulting in marginal failures and leakage. The current development of composites whose polymerization occurs by the opening of the rings rather than by free radical polymerization and approximation of dimethacrylate monomers has evidenced a reduction in polymerization shrinkage. Silorane-based resin composites have their organic matrix formed by siloxane and oxirane with cationic polymerization and opening of the rings during polymerization reaction [14, 24]. This has resulted in low polymerization shrinkage, insolubility to simulated biological fluids, and good color stability when compared with dimethacrylate-based composites s [5, 7, 10, 24]. Despite these advantages, further studies are necessary to evaluate the behavior of silorane-based resin composites in cases requiring repair.

During the clinical life of a restoration, failures can occur, which leads to the indication of repair instead of its total replacement. Clinical researches on repairing, reshaping and sealing of direct restorations have demonstrated that these minimal intervention procedures are conservative alternatives indicated in cases of failure in marginal adaptation and staining [13, 21]. Therefore, when properly indicated, the repair procedure has been considered as an alternative to total replacement of a restoration [6] decreasing the weariness of the sound tooth structure and the time amount required to remove the restorative material completely [16].

Notwithstanding, the repairing requires that the restoration surface is adequately treated [1, 9]. Among these treatments, silanization has shown controversial results, with either effectiveness in [22] or unable of increasing the bond strength [3]. On the other hand, the surface roughness has exhibited good results [9], probably because of promoting a micromechanical linking between the old resin composite and the new restorative system.

The superficial treatments for repairing most taught in dental schools have been the roughening of the exposed surface, etching with phosphoric acid, cleaning with pumice and abrading with aluminum oxide [1]. The materials most used in repairing have been the adhesive agents, resin composite, silane agent, and flowable resin [1]. However, most of the studies were conducted with resins containing organic matrixes with similar chemical characteristics [9, 11].

The concern with the compatibility between resins with different organic matrix, such as silorane-based and conventional resins, is clinically relevant for eventual repairs because not even the dentist knows the composite type employed firstly [4]. The surface weariness followed by silorane-based bonding agent has been recommended to repair silorane-based resins with similar resins while the application of the primer agent has been contraindicated [17]. On the other hand, when a dimethacrylate-based resin is used in the repair, an application of an intermediary layer of silorane-based bonding agent has been advised [12, 23].

Dimethacrylate-based material bond strength to silorane-base materials has been little studied probably because the latter is recently on the market. Consequently, both the repairing effectiveness and which would be the best surface treatment has not been clarified yet. Moreover, the behavior of the interaction between the different layers should be evaluated at long-term, which can be simulated by laboratorial studies.

The aim of this study was to evaluate the microshear bond strength of the repair interface between a silorane-/silorane-based resin as well as a silorane-/dimethacrylate-based resin.

Material and methods

Eighty matrixes made from acrylic resin (Clássico, São Paulo, SP, Brazil) within PVC tubes (3/4” in diameter, 1 cm in height), with a center cylindrical orifice with 5 mm in diameter and 3 mm in mean depth. The restorative materials and the adhesive agents employed are described in table I.

Table I – Composition of the materials employed

Material, batch, shade	Manufacturer	Composition
Filtek P90 80284930218 A3	3M ESPE	Particles of quartz and silica/silane, yttrium fluoride, Di-3-4-epoxy cyclohexyl dimethyl silane, 3-4 poly methyl siloxane-epoxy-cycle, functional di- and oligo-siloxane, initiator system: camphorquinone and iodonium salt (donator of eletrons), stabilizers and pigments.
Filtek Z350 XT 80284930218 A3B	3M ESPE	Bis-GMA (bisphenol A glycidyl methacrylate), UDMA (urethane dimethacrylate), TEGDMA (triethylene glycol dimethacrylate), bis-EMA (bisphenol A ethoxylated dimethacrylate), particles of silica and zirconia/silane, BHT (Butyl hydroxy toluene), photoinitiator system and pigments.
Silorane adhesive system – Self-Etch Primer	3M ESPE	Phosphate methacrylates, copolymer of Vitrebond, BisGMA, HEMA (hydroxy-ethylene glycol dimethacrylate), water, ethanol, particles of silica treated with silane, initiators, and stabilizers.
Silorane adhesive system – Bond	3M ESPE	Hydrophobic dimethacrylate, phosphate methacrylates, TEGDMA, particles of silica treated with silane, initiators, and stabilizers.
Single Bond	3M ESPE	BisGMA, HEMA, dimethacrylates, ethanol, water, photoinitiator system, functional copolymer of methacrylate of polyacrylic and polyalkenoic acids.

The silorane-based resin composite (Filtek P90, 3M ESPE, St. Paul, USA) was inserted within the orifice of acrylic resin matrix with the aid of a spatula, at two increments, in order to obtain a base disc as substrate for the silorane-based resin composite. Each increment (1.5 mm in thickness) was individually light-cured for 40 s with the aid of a light-curing unit (Coltolux LED, Coltène/Whaledent, Altstätte, Switzerland), at intensity of 1,000 mW/cm^2. After the insertion of the last increment of resin composite P90, a polyester strip (TDV Dental Ltda., Pomerode, SC, Brazil) was placed onto the surface of the resin not polymerized and pushed with the aid of a glass lamina with 1 mm in thickness to achieve a flat surface prior to photoactivation, which was carried out through the polyester strip. To obtain the aging of the resin composite specimens prior to the repairing procedures, the discs were stored into distilled water at 37°C for one week. This was executed to mimic the condition of a restoration exposed to the humidity within oral cavity.

The discs were divided into six groups (G1 to G6). The specimens from G1, G3 and G5 were repaired with a silorane-based material (Filtek P90), while those from G2, G4 and G6 were repaired with a dimethacrylate-based material (Filtek Z350 XT, 3M ESPE, St. Paul, USA). The repairs were executed after the washing of the discs with air jet for 15 s and drying with air jet for 20 s at 10 cm of distance. G5 and G6 specimens were roughened with the aid of 600-grit sandpaper, at a single direction with water lubrication (Ecomet 250, Lake Buff, IL, USA), prior to the treatment with the adhesive systems and repairing procedures.

The specimens of G4 and G6 underwent 37% phosphoric acid etching (Dentak Villevie, Joinville, SC, Brazil) for 30 s. G6 specimens received silane application (ProSil, FGM, Joinville, SC, Brazil), while the adhesive system Single Bond 2 (3M ESPE, St. Paul, USA) was applied onto the specimens of G4 and G6 prior to the repairing procedure with dimethacrylate-based resin (Z350 XT). The adhesive system Single Bond 2 application was performed with the aid of a microbrush, twice, followed by gentle air drying for 5 s and light-curing for 10 s.

The specimens of G3 and G5 were washed and dried before the treatment with the adhesive system P90 (3M ESPE, St. Paul, USA) and the repairing procedure was executed with silorane-based material (P90). The application of the two-flask adhesive system P90 (Self-Etch Primer and Bond agent) was executed according to the manufacturer's recommendation. The self-etch primer agent was applied with the aid of a microbrush for 15 s, followed by gentle air drying and light-curing for 10 s. Table II exhibited the description of the experimental groups.

Table II – Groups and treatments prior to either repairing procedure (G1 to G6) or immediate adhesion (G7 and G8)

Groups	Aging	600-grit sandpaper roughness	Washing for 15 s, drying for 20 s	37% phosphoric acid etching	Silane	Adhesive	Repairing resin
G1	1 week into water at 37°C	No	Yes	No	No	No	P90
G2	1 week into water at 37°C	No	Yes	No	No	No	Z350
G3	1 week into water at 37°C	No	Yes	No	No	P90	P90
G4	1 week into water at 37°C	No	Yes	Yes	No	Single Bond	Z350
G5	1 week into water at 37°C	Yes	Yes	No	No	P90	P90
G6	1 week into water at 37°C	Yes	Yes	Yes	Yes	Single Bond	Z350
G7	No	No	No	No	No	No	P90
G8	No	No	No	No	No	No	Z350

Cylindrical silicone tubes (Perfitécnica, Salto, SP, Brazil) with 0.95 mm in internal diameter and 1.5 mm in height were used as matrixes for either the immediate adhesion or repairing procedure after the surface treatment of each silorane disc, according to the group assigned (n = 10). One portion of resin composite was inserted into the cylindrical silicone tube with the aid of a spatula. After the cleaning of the tube extremities with the same spatula, the set was put in contact with the center of the silorane disc with the aid of dental tweezers. The photoactivation of the resin within the cylindrical tube in contact with the disc enabled their adhesion therefore simulating the execution of a repairing procedure, but with standardized characteristics. Elapsing 10 minutes, the silicone tube was cut with the aid of a size 15 blade to expose the resin cylinder and the repairing interface. Following, the specimens were stored into distilled water at 37°C.

Cylinders of silorane- (Filtek P90) and dimethacrylate-based (Z350 XT) materials were constructed directly onto the discs of G7 and G8, respectively, without previous aging, to analyze the immediate adhesion between the materials.

Thus, 80 specimens were submitted to micro shear bond strength test through a universal testing machine (EMIC, model DL3000, São José dos Pinhais, PR, Brazil) and observed regarding to failure types. The acrylic resin matrixes were mounted into a round device and fixed to the testing machine with load applied with the aid of a steel wire (0.2 mm in diameter) and crosshead speed of 0.5 mm/min until rupture.

The bonding strength was calculated with the aid of software linked to the testing machine by dividing the maximum force recorded (in Newton) by the bonding area (in mm^2), and expressed in MPa. After the shear bond strength test, the specimens were examined with the aid of stereoscopic magnifying glass at x30 magnification (SZX9, Olympus, Tokyo, Japan), to determine the failure mode, which were classified as follows: adhesive (at the bonding interface), cohesive (at the base resin or cylinder resin) and mixed. The data was submitted to statistical analysis through Student's t test with level of confidence of 95%.

Results

Micro shear bond strength values indicated an increasing in the bonding strength of the repairing procedures with roughening and application of the adhesive system onto the base disc (P90). Moreover, the results showed in table III and figure 1 demonstrated that the differences among G1, G3, G4, G5, G6 and G7 were not statistically significant ($p < 0.05$). The greatest micro shear bond strength mean values were observed in the specimens of G7 with immediate adhesion between silorane-/silorane-based resins without previous aging.

Table III – Micro shear bond strength means and standard deviations (MPa) of the different groups

Groups	Bond strength (MPa)
G1	13.71 ± 8.67 ^{a}
G2	4.09 ± 3.87 ^{b}
G3	15.41 ± 8.91 ^{a}
G4	11.41 ± 6.82 ^{a}
G5	19.33 ± 5.29 ^{a}
G6	14.97 ± 3.69 ^{a}
G7	21.28 ± 5.90 ^{a}
G8	1.52 ± 1.86 ^{b}

Superscript letters mean statistically significant differences ($p < 0.05$)

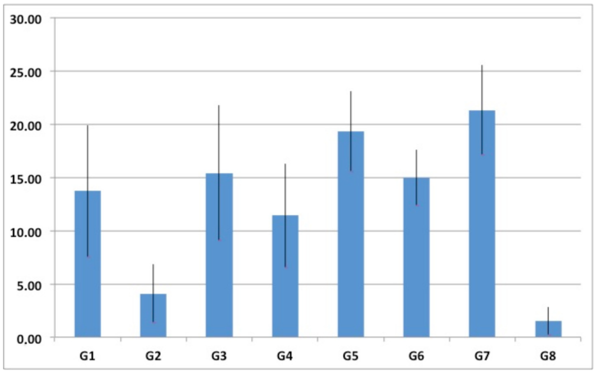

Figure 1 – Micro shear bond strength of the different groups (MPa, means and standard deviations). G1 to G6 received repairing procedures (G1, G3 and G5 with P90; G2, G4 and G6 with Z350). G1 and G2 did not receive surface treatment, G3 and G4 received adhesive system; G5 and G6 received roughening procedure and adhesive system. G7 and G8 received immediate adhesion without repairing with either P90 or Z350, respectively

The smallest micro shear bond strength mean values were observed in the specimens of G8 and G2, that is, in the repairing procedure between silorane-/dimethacrylate-based material without surface treatment and in the immediate silorane-/dimethacrylate-based material respectively. Significantly highest micro shear bond strength values were observed in groups in which the surfaces had been treated with adhesive systems (G5 and G6), both for repairing procedures with silorane (G3 and G5) and dimethacrylate (G4 and G6), but without statistically significant differences when compared with G7 (immediate adhesion between silorane-/silorane-based materials). The decreasing of the adhesivity after the aging period of the material surface was observed by comparing G7 with G1, without statistically significant differences.

Figures 2 and 3 exemplified the failure types seen with the aid of the stereoscopic magnifying glass at x30 magnification. Cohesive failures were observed when part of the material within the cylinder was retained onto the base resin (figure 3B). The highest micro shear bond strength mean values (table III), exhibited by the immediate adhesion between silorane-/silorane-based materials (G7), and corresponded to cohesive failures. All failures of the immediate adhesion between silorane-/dimethacrylate-based materials (G8) and of the repairing procedure between silorane-/dimethacrylate-based materials without surface material (G2) were adhesive type (figure 2). In the groups submitted to the surface treatment with adhesive system and roughening, both for silorane-/silorane-based materials and silorane-/dimethacrylate-based materials, showing bond strength mean values significantly higher than those of G2 and G8, exhibited mixed and cohesive failures.

In mixed failures, fragments of the cylinder resin were retained into the base resin (figure 3C). All failures within G2 and G8 were adhesive failures (figure 2), with the smallest micro shear bond strength mean values (figure 1). Adhesive failures where the base resin (substrate of resin composite P90) were clean and smooth, without resin remnants of the cylinder resin (figure 3D).

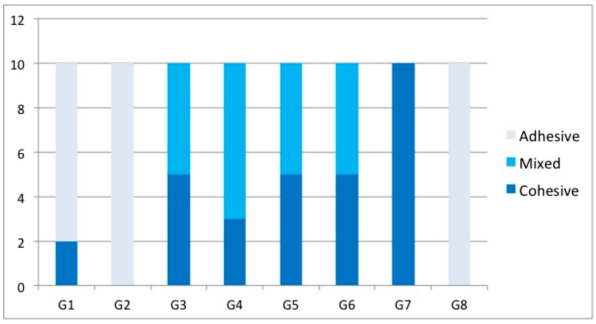

Figure 2 – Percentage (%) of the failure types during the micro shear bond strength testing (n = 10, per group)

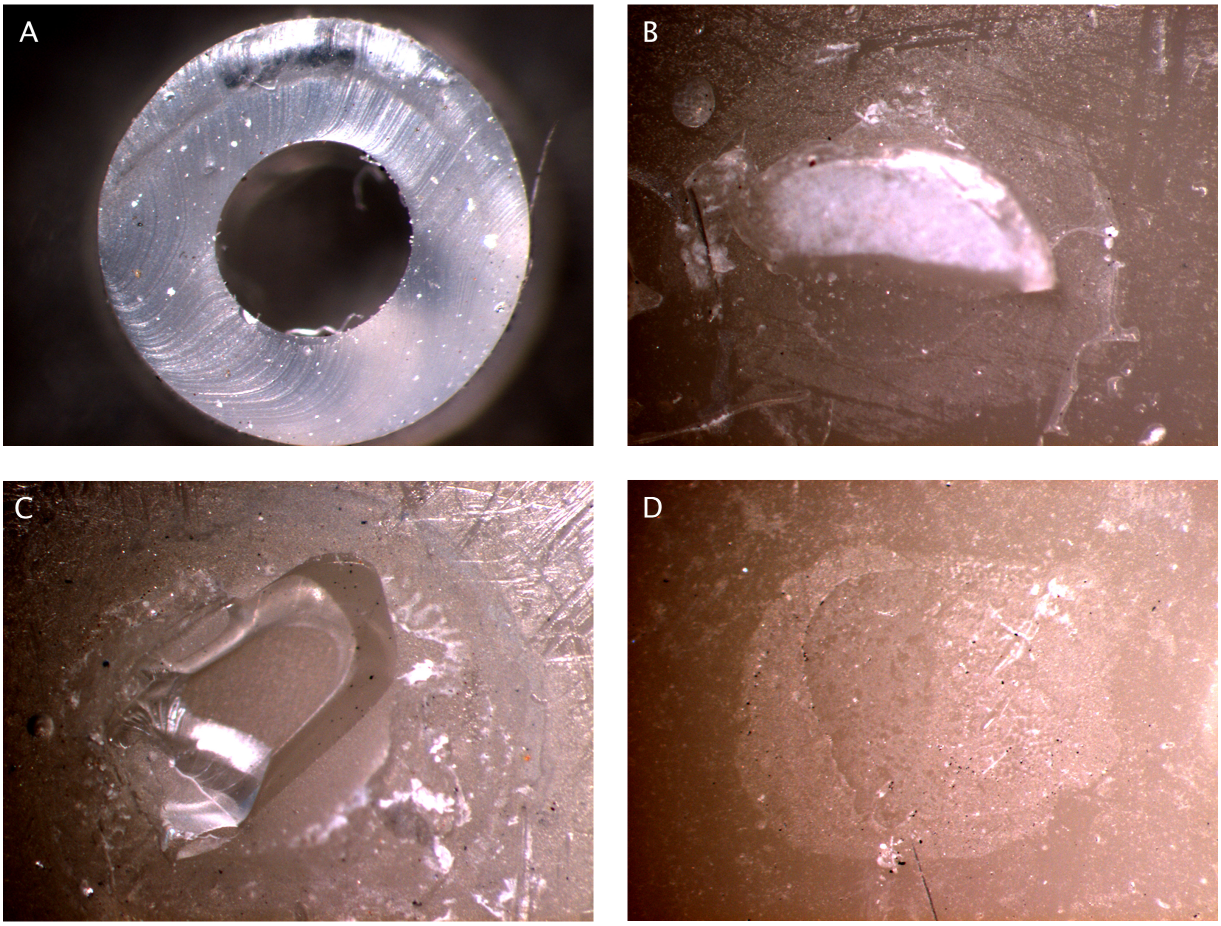

Figure 3 – Silicone cylinder and disc surfaces after the micro shear bond strength testing (x30 magnification): (a) empty cylinder to show its internal area to be filled with the resins to simulate the repairing procedure (0.95 mm in internal diameter); (b) cohesive failure; (c) mixed failure; (d) adhesive failure

Discussion

By considering the different chemical characteristics between the dimethacrylate- and silorane-based resin composite, the aim of this study was to evaluate the bond strength between them. In the dimethacrylate-based composites, the oxygen-inhibited layer formed onto the surface after the polymerization of the material increments in contact with the environmental air enables a better adhesion between the layers of these materials [8]. On the other hand, the cationic polymerization of silorane-based materials do not lead to the formation of oxygen-inhibited layer and the decreasing of adhesion among successive increments of silorane over time [23] would cause the decreasing of micro shear bond strength. For this reason, in this present study, the preparation of each specimen of G7 (control group – immediate adhesion between silorane/silorane) was executed with less than 5 min, to assure the greatest reactivity among successive increments, as suggested by Tezvergil-Mutluay *et al.* [23], who observed the greastet immediate adhesion values of silorane increments when those were inserted and light-cured in few seconds [23]. The greatest micro shear bond strength values were observed in the specimens of G7, as cylinders of silorane-based composites were made immediately onto discs of the same resin, therefore enabling cohesive failures within all specimens.

As discussed above, the chemical reactivity of the components of silorane-based composites accounting for the adhesion of successive increments decreases over time and could affect the adhesion of repairing procedures [23]. Notwithstanding, in this present study, the reduction of the adhesivity after the aging of the material surface was not statistically significant when the specimens submitted to immediate adhesion between silorane-/silorane-based material (G7) were compared with those underwent repairing without surface treatment (G1). These results demonstrated that despite of the aging period and conditions, the material still has enough reactivity to provide adhesion with chemically-compatible repairing material.

The smallest micro shear bond strength mean values were seen in G8 specimens submitted to immediate adhesion between silorane-/dimethacrylate-based materials. Other authors still verified a smaller immediate bond strength between silorane-/dimethacrylate-based materials, without either aging period or surface treatment [23]. The result of the lack of adhesion between the dimethacrylate-based and freshly-light-cured silorane-based material when this exhibits the greatest chemical reactivity confirms the lack of chemical affinity between the materials.

This present study used a one-week period into distilled water at 37°C as aging method. Different authors studying repairing procedures on silorane-based resins have employed different techniques to mimic the conditions to which the restoration are submitted within oral cavity: 72 hours into saline solution at 37°C [12], 24 hours into water at 100ºC [15], one month into water at 60°C [18], and 24 hours into water at 37°C [20]. These methods have been used to promote the water sorption and leaching of components not participating in the polymerization reaction, prior to the repairing procedure.

Micro shear bond strength testing was chosen in this study because it does not require additional cuts after the bonding of the segments during the preparation of the specimens, taking into consideration the low bond strength observed by other authors in both the immediate adhesion and the repairing between silorane- and dimethacrylate-based resins [4, 12, 23]. The evaluation method of micro shear bond strength through orthodontic wire has been suggested as an alternative to microtensile bond strength because of its easier execution and good distribution of stresses in the adhesive interfaces [2].

According to Tezvergil-Mutluay *et al.* [23], the interposition of silorane-based adhesive system as intermediary layer would provide a better immediate adhesion between silorane-/dimethacrylate-based materials than that provided by dimethacrylate-based adhesive systems. The initial adhesion between silorane-/dimethacrylate-based materials thorugh silorane adhesive system, composed by dimethacrylate with carboxylic and phosphoric acid groups, enables the reaction of the phosphate group with oxirane and that of acrylate with dimethacrylate promoting chemical adhesion [12, 23]. The effect of the lack of chemical affinity between silorane and dimethacrylate impairing the initial adhesion (G8) was not decreased by the surface aging of G2, but it occurred with the application of dimethacrylate-based adhesive system (G4). In the treatment with the exclusive application of the dimethacrylate-based adhesive system (G4) prior to repairing procedure, the micro shear bond strength values were not statistically different from those of the initial silorane-silorane adhesion (G7). The effect of dimethacrylate-based adhesive system application was more substantial than the decreasing of silorane chemical reactivity during the aging period, with statistically significant difference between G3 and G8.

In G5 and G6, where the repaired surfaces were treated with roughening prior to the application of the adhesive systems, bond strength values were not statistically different from those of the immediate silorane/silorane adhesion (G7), both repaired with silorane- (G5) and dimethacrylate-based resins (G6). The roughening of material surface to be repaired promoted the weariness and development of micro rugosities. The intermediary interfaces created by roughening and the filling with low viscosity material provide micro retentions and allow a better adaptation with the repairing material of high viscosity. The roughening obtained with the aid of 600-grit sandpaper, employed in this present study, was also used by other authors [12]. Other roughening methods have been used in silorane repairing: 80-grit sandpaper [18], 320-grit sandpaper [15], abrasion with diamond drill and aluminum oxide blasting [25].

The surface treatment for the repairing between silorane-/dimethacrylate-based materials with roughening but without silane application prior to the application of the dimethacrylate adhesive system was not analyzed by this present study. The silane application executed in the repairing procedure of G6 could have contributed to increase the micro shear bond strength values as observed by other authors who used silane-based adhesive system [18] and silane prior to the adhesive system [25], which suggested that the silane promoted a greater superficial leakage.

The greatest micro shear bond strength mean values observed in G7 (table III) corresponded to cohesive failures (figure 2). In mixed failures, fragments of the cylinder resin were retained into the base (figure 3C). The failures in G2 and G8 were of adhesive type (figure 2), with the smallest micro shear bond strength mean values (table III). In adhesive failures, the silorane discs were clean and smooth, without fragments of the cylinder resin (figure 3D). These results suggested that there would be little or none adhesion between the two resins.

In this present study, adhesive systems indicated for either P90 and dimethacrylate, considering the two possible clinical situations: the composition of the original resin to be repaired is either known or unknown. Although this present study did not verify significant differences between the roughening treatment followed by the adhesive system and the single application of the adhesive system, roughening seems to be a more reliable surface treatment for repairing procedures because it increases the adhesion area.

Conclusion

Surface treatments with the application of either adhesive systems or roughening enabled the repairing procedures in silorane-based resin composites when an adhesive system compatible with the repairing material was used. These results suggested that repairing procedures onto silorane-based resin composites can be executed successfully, regardless of the surface roughening and the composition of the repairing material.

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