Effect of Surface Treatments and Bonding Agents on the Bond Strength of Repaired Composites

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ABSTRACT

Statement of the Problem: An adequate repair procedure depends on high bond strength between the existing composite and the new composite.

Purpose: To evaluate the effect of surface treatments and bonding procedures on the bond strength of repairs performed 24 hours after composite polymerization.

Materials and Methods: Composite specimens were stored in distilled water at 37°C for 24 hours. Specimens were allocated into 12 groups ($N = 10$) according to the combination of surface treatment (none, air abrasion, diamond bur) and bonding procedure (none, Single Bond after H_3PO_4 cleansing, Clearfil SE Bond after H_3PO_4 cleansing, Clearfil SE Bond without H_3PO_4 cleansing). The ultimate tensile strength (UTS) of the composite was tested in nonrepaired specimens. Twenty-four hours after repair, specimens were sectioned into three slabs and trimmed to an hourglass shape (1 mm^2 area). Slabs were tested under tension and mean bond strengths analyzed with two-way analysis of variance/Tukey and Dunnett tests ($\alpha = 5\%$).

Results: Two groups resulted in repair bond strengths similar to composite UTS: air abrasion combined with Clearfil SE Bond after H_3PO_4 cleansing, and air abrasion combined with Clearfil SE Bond without H_3PO_4 cleansing. Combinations of surface treatments and bonding procedures were not statistically different.

Conclusions: When repair procedure was performed 24 hours after composite polymerization, different combinations of surface treatments and bonding procedures affected repair bond strength similarly. There was no statistical difference between the repair bond strength of groups air-abraded and bonded with the self-etching system and composite UTS.

CLINICAL SIGNIFICANCE

Only air abrasion associated with a self-etching system provided repair bond strength comparable to composite UTS.

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INTRODUCTION

Composites have been widely used for direct restorations. However, failures or fractures involving restorative materials might occur, which can lead to clinical problems.[1–7] The complete removal of defective composite restorations is not always necessary or desirable because restoration replacement can damage tooth structure, creating even wider preparations.[7–10] For this reason, the repair of a restoration, instead of its removal, would be a favorable procedure.[2–7,9,11–13]

Bonding between two composite layers is accomplished by the presence of an oxygen-inhibited layer of unpolymerized resin.[4,14] However, if the composite has been contaminated, polished, processed in a laboratory, or aged, the adhesion to a new composite might not be accomplished adequately.[3,15] Various methods have been suggested to establish an adequate bond strength between the existing composite and the new composite.[2,3,5–7,9,12,14,15] These methods include surface treatments and the use of intermediate bonding agents to enhance repair bond strength.[2,3,5,6,9,15–19] While surface roughness promotes mechanical interlocking, the bonding agent improves surface wetting and chemical bonding with the new composite.[2] However, there is no consensus on what protocol would be more successful for composite repair.[3,6,15] Repair protocols have shown widely variable repair bond strengths, which are in the range of 25 to 80% of the cohesive strength of the substrate material.[12,15,18,20,21]

Self-etching adhesive systems were developed to simplify adhesion procedures. An advantage attributed to these systems is the elimination of sensitive steps such as acid-etching, rinsing, and dentin drying.[22–24] Self-etching systems can be used to condition both the surrounding tooth and the composite to be repaired in one procedure.[20] Recently, the effectiveness of those systems in repairing composites aged 6 years was investigated.[20] The authors observed that the repair of aged composites seems to be feasible with the use of self-etching systems. High repair bond strengths were found with a two-step self-etching system, and this finding was justified by an efficient capacity to wet the composite surface.[20]

This study was designed to evaluate the effect of different surface treatments and bonding systems on the bond strength of composite repairs. Two null hypotheses were tested: (1) different combinations of surface treatments and bonding procedures do not affect the repair bond strength; and (2) repair bond strengths are not as high as the ultimate tensile strength (UTS) of the composite.

MATERIALS AND METHODS

The restorative materials used in the present study, along with their classification, manufacturers, batch numbers, and composition, are given in Table 1.

One hundred ten composite specimens were made from a hybrid composite containing a bisphenol-A-glycidyl ether dimethacrylate (Bis-GMA) matrix (TPH Spectrum, Dentsply DeTrey, Konstanz, Germany). A quadrangular mold (5.0 mm × 5.0 mm × 3.0 mm dimension) was filled with two increments of the composite (1.5 mm each) (Figure 1). After the insertion of the last increment, a Mylar strip and a 500-g weight were placed over the mold and left for 30 seconds to allow for a better accommodation of the composite. Each increment was light-cured for 20 seconds using a halogen light-curing unit (Optilux 501, Sybron Kerr, Danbury, CT, USA). The light output of the light-curing unit was measured with its radiometer and was greater than 500 mW/cm^2. Specimens received an identification number and were stored individually in 6 mL of distilled water at 37°C for 24 hours. The surface directly exposed to the visible light was identified and the repair procedure was performed over this surface.

Ten additional specimens were prepared using a quadrangular mold with different dimensions (5.0 mm

TABLE 1. DESCRIPTION OF RESTORATIVE MATERIALS USED IN THE PRESENT STUDY, ALONG WITH THE BATCH NUMBERS, MANUFACTURER, AND COMPOSITION.

Material/Batch Number	Manufacturer	Composition
Scotchbond Etchant gel/3BH	3M ESPE, St. Paul, MN, USA	35% phosphoric acid, silicon dioxide
Single Bond/3JC	3M ESPE	Bis-GMA, HEMA, PAA, photoinitiators, ethanol, water
Clearfil SE Bond/Primer: 00410A; Bond: 00547A	Kuraray Co., Osaka, Japan	SE Primer: N,N-Diethanol-p-toluidine, MDP, HEMA, hydrophilic dimethacrylate, DL-camphorquinone, water SE Bond: N,N-Diethanol-p-toluidine, MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, DL camphorquinone, silanated colloidal silica
TPH Spectrum/Shade A1:22162; Shade C2:16168	Dentsply DeTrey, Konstanz, Germany	Bis-GMA, Bis-EMA, TEGDMA, barium aluminoborosilicate, silica

Bis-EMA = ethoxylated bisphenol-A-dimethacrylate; Bis-GMA = bisphenol-A-glycidyl ether dimethacrylate; HEMA = 2-hydroxyethyl methacrylate; PAA = polyalkenoic acid copolymer; MDP = 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA = triethylene glycol dimethacrylate.

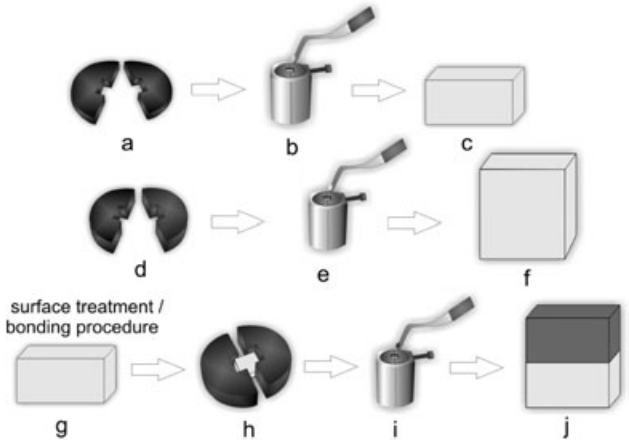

Figure 1. A, *Quadrangular mold—5 × 5 × 3-mm dimension.* B, *Insertion of the composite.* C, *5 × 5 × 3-mm specimen.* D, *Quadrangular mold—5 × 5 × 6-mm dimension.* E, *Insertion of the composite.* F, *Nonrepaired specimens.* G, *Specimen after the respective surface treatment/bonding procedure.* H, *Specimen inserted in the 6-mm-high mold.* I, *Mold filled with the repair composite.* J, *Repaired specimen.*

× 5.0 mm × 6.0 mm). This mold was filled with four increments of the composite (1.5 mm each). After the insertion of the last increment, the Mylar strip and the weight were placed over the mold and left for 30 seconds, and the specimen was light-cured through the strip for 20 seconds. These specimens were fabricated to test the UTS of the composite resin (Figure 1).

After 24 hours, the specimens of the 5.0 mm × 5.0 mm × 3.0 mm dimension were randomly allocated into 11 experimental groups (N = 10) according to the combination of surface treatment and repair bonding procedure. Experimental groups are listed in Table 2. Directions for surface treatments and bonding procedures are described in Table 3.

For the repair procedure, the 6.0-mm-high quadrangular mold was used. After the respective surface treatment and bonding procedure, each specimen was inserted in the mold leaving a 3.0-mm space to be filled by the repair composite

TABLE 2. EXPERIMENTAL GROUPS.

Group	Surface Treatment	Bonding Procedure
1	None	Phosphoric acid cleansing; Single Bond application
2	None	Phosphoric acid cleansing; Clearfil SE Bond application (SE Primer + SE Bond)
3	None	Clearfil SE Bond application (SE Primer + SE Bond) without previous acid cleansing
4	Air abrasion with 50 µm Al_2O_3 particles*	None
5	Air abrasion	Phosphoric acid cleansing; Single Bond application
6	Air abrasion	Phosphoric acid cleansing; Clearfil SE Bond application (SE Primer + SE Bond)
7	Air abrasion	Clearfil SE Bond application (SE Primer + SE Bond) without previous acid cleansing
8	Roughen with diamond bur†	None
9	Roughen with diamond bur	Phosphoric acid cleansing; Single Bond application
10	Roughen with diamond bur	Phosphoric acid cleansing; Clearfil SE Bond application (SE Primer + SE Bond)
11	Roughen with diamond bur	Clearfil SE Bond application (SE Primer + SE Bond) without previous acid cleansing
12	Control—ultimate tensile strength of the composite	

*Microetch Bioart, São Carlos, SP, Brazil.
†3098, KG Sorensen, Barueri, SP, Brazil.

TABLE 3. DIRECTIONS FOR SURFACE TREATMENTS AND BONDING PROCEDURES.

Surface Treatment	Directions for Use
Air abrasion	The abrasion unit was positioned at 5.0 mm from the surface. The surface was abraded for 10 seconds (pressure of 60 psi), rinsed with distilled water, and dried with oil-free compressed air.
Roughen with diamond bur	The surface was slightly roughened with a coarse diamond bur rotating at high speed with constant water spray. A cylindrical bur roughened the composite surface for 3 seconds.
Bonding Procedure	**Directions for Use**
Scotchbond Etchant, phosphoric acid	Surfaces were cleaned with 35% phosphoric acid gel (30 seconds), rinsed with distilled water (15 seconds), and dried with oil-free compressed air.
Single Bond adhesive system	Adhesive was applied with two consecutive layers, air dried, and light-cured (10 seconds).
Clearfil SE Bond adhesive system	SE Primer was actively applied (20 seconds) and dried with mild air flow; SE Bond was applied, gently air dried, and light-cured (10 seconds).

(Figure 1). The repair composite was incrementally inserted and light-cured as previously described. A dark shade was selected as repair material (C2) and a lighter one as substrate (A1) to provide a better assessment of the repair interface.

Twenty-four hours after the repair procedure, the experimental units were prepared for a microtensile bond test (Figure 2). Specimens

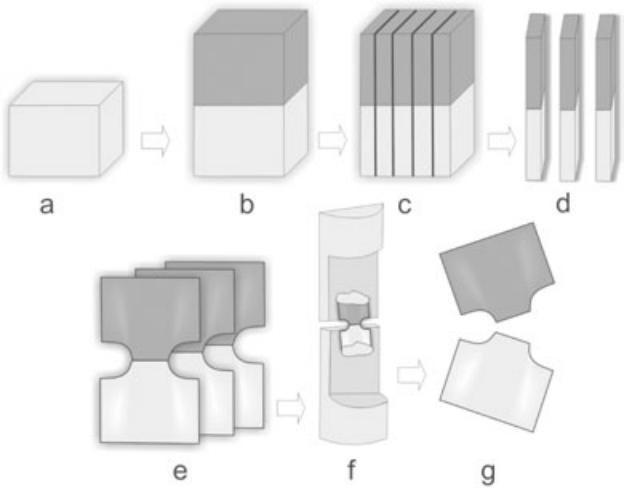

Figure 2. A, *Specimen before repair procedure.* B, *Repaired specimen.* C, *Sliced specimen.* D, *Three slabs to be tested.* E, *Slabs with an hourglass shape.* F, *Hourglass in the microtensile apparatus.* G, *Fractured specimen.*

were sectioned perpendicular to the bonded repair interface into 1-mm-thick slabs ($N = 3$ per specimen) with a low-speed diamond saw (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA) under constant water refrigeration. Each slab was trimmed along the repair interface to an hourglass shape using a super-fine diamond bur (1090FF, KG Sorensen, Barueri, SP, Brazil) in a high-speed handpiece under air/water spray refrigeration. These procedures yielded bonded surface areas of approximately 1 mm^2. The number of slabs prematurely debonded during specimen preparation was recorded, and spontaneous debonds were considered as 0 MPa.

Specimens were attached to the flat grips of a microtensile bond strength testing device with cyanoacrylate glue (SuperBonder Gel, Henkel Loctite, Itapevi, SP, Brazil) and tested under tension in a universal testing machine (Instron 4411, Instron Corporation, Canton, MA, USA) at a crosshead speed of 0.5 mm/min until failure. After failure, each adhesive interface area was measured and the bond strength value (MPa) was calculated.

The bond strength of each specimen was obtained from the arithmetic mean of its three slabs. A two-way analysis of variance analyzed bond strength data with main factors "surface treatment" and "bonding procedure." Multiple pairwise comparisons were performed with Tukey post-hoc test. The Dunnett test was used to compare the UTS of the composite with the bond strength obtained in repaired groups. Statistical analyses were performed using SAS 8.0 for Windows (SAS Institute, Cary, NC, USA) at a significance level of 5%.

After the μTBS testing, the fracture mode of each repair technique was evaluated using a stereoscopic microscope (×45, Meiji 2000, Meiji Techno, Saitama, Japan). The slabs were classified according to their main characteristics[3]: type 1—adhesive failure between composite/repair interface; type 2—mixed failure (association of more than one type of failure); and type 3—totally cohesive composite failure (substrate or repair).

RESULTS

Bond strengths and standard deviations are listed in Table 4. There was a significant interaction between main factors: "surface treatment" × "bonding procedure" ($p = 0.019$). When no adhesive system was used, surface treatments (air abrasion and diamond bur) presented similar mean bond strengths; both had repair bond strength significantly lower than the composite UTS. Also, groups with bonding procedures and without surface treatment presented similar means, which were inferior to those observed for the composite UTS. Combinations of surface treatments (air abrasion and diamond bur) and bonding procedures (H_3PO_4 +

TABLE 4. BOND STRENGTH VALUES (MPA), STANDARD DEVIATIONS, NUMBER OF SLABS PREMATURELY FAILED (PF), AND MEAN BOND STRENGTH EXPRESSED AS PERCENTAGE OF THE ULTIMATE TENSILE STRENGTH (UTS).

Surface Bonding Procedure / Treatment	None	H_3PO_4 + Single Bond	Clearfil SE Bond	H_3PO_4 + Clearfil SE Bond
None	43.8 (7.2) Aa pf = 2 UTS (100%)	31.7 (7.6) Ab* pf = 4 72.4%	29.3 (8.0) Ab* pf = 8 66.9%	30.9 (9.5) Ab* pf = 3 70.5%
Air abrasion	33.3 (12.5) Ba* pf = 8 76%	33.8 (7.1) Aa* pf = 5 77.1%	36.0 (11.5) Aa pf = 3 82.2%	36.0 (8.6) Aa pf = 4 82.2%
Roughen with diamond bur	31.4 (5.2) Ba* pf = 2 71.6%	31.7 (4.0) Aa* pf = 2 72.4%	31.0 (13.4) Aa* pf = 3 70.7%	28.4 (7.2) Aa* pf = 2 64.8%

*Means statistically different from the UTS of the composite (Dunnett test, $p < 0.05$).
Means followed by different letters indicate statistical significant difference (two-way analysis of variance/Tukey test, $p < 0.05$). Upper case letters compare "surface treatments" within each "bonding procedure." Lower case letters compare "bonding procedure" within each "surface treatment."

Single Bond; Clearfil SE Bond and H_3PO_4 + Clearfil SE Bond) were not statistically significant.

Two repaired groups presented repair bond strength similar to the composite UTS: air abrasion combined with Clearfil SE Bond after H_3PO_4 cleansing and air abrasion combined with Clearfil SE Bond without H_3PO_4 cleansing.

Figure 3 shows the distribution of the failure modes among experimental groups. Only cohesive failures (type 3) were observed in the nonrepaired group (composite UTS). Predominantly, cohesive failures were not found in any other group; only adhesive (type 1) and mixed (type 2) failures were observed.

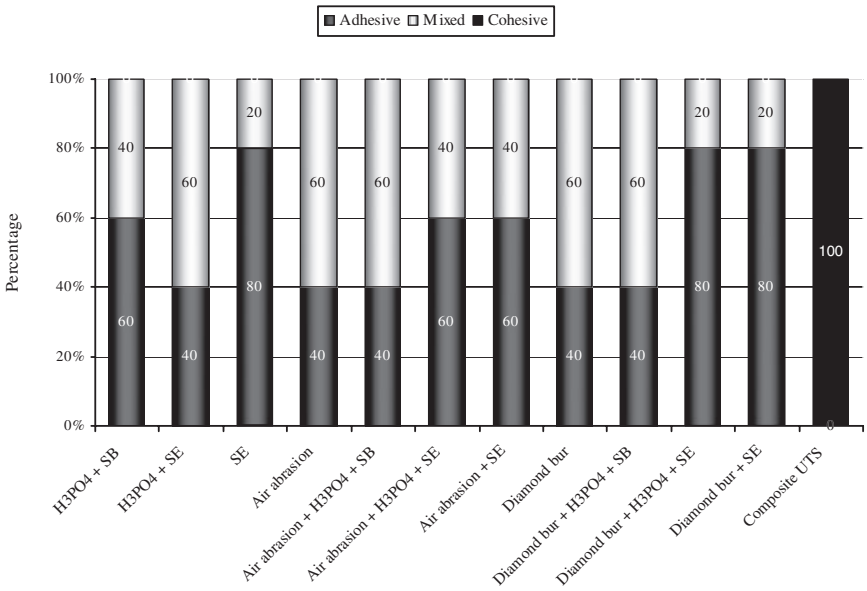

Figure 3. Distribution (percentage) of failure mode.
SB = single bond, SE = Clearfil SE bond, UTS = ultimate tensile strength.

DISCUSSION

Repairs are alternatives for correcting some failures in composite restorations. For a successful repair, an adequate bonding interface between the existing resin and the new one must be created. This interface must provide bond

strength similar to the cohesive strength of the substrate composite. Thus, the possibility of failures at the composite–repair interface, such as fractures or microleakage, is diminished. In the present study, different immediate composite repair approaches were evaluated, and the microtensile bond testing was used to measure the repair bond strength. One advantage of the microtensile test is that the bonded interface of small specimens has a better stress distribution during loading.[25] This test often results in higher apparent bond strengths than those found in tests using large specimens.[25]

The surface roughness of the composite to be repaired appears to have a significant influence on repair bond strength.[2,3,9,12,15,26] Air abrasion is a surface treatment that causes "micro" retentive features, while a diamond stone yields "macro" and "micro" retentive features. Without a bonding system, greater bond strength is expected from devices yielding macro retentive features.[2] On the other hand, with bonding agents, a better surface wetting occurs as the adhesive resin infiltrates into the composite microscopic surfaces.[2,6,9]

In the present study, repair bond strengths ranged from 67 to 82% of the UTS of the composite. There was a statistical interaction between the main factors (surface treatment × bonding procedure), indicating that the composite UTS could not be accomplished with only one repair approach (surface treatment or bonding procedure). Surface-treated groups and groups with untreated surfaces presented similar repair bond strengths, irrespective of the bonding procedure. These results are not in agreement with previous reports, which stated that the combination of surface treatments and bonding systems increases the repair bonding significantly.[2,6,9] However, these findings could be explained by a possible chemical interaction between the resin-based materials (substrate and repair).[27]

There are some clinical situations that may require the repair of a restoration 24 hours after its placement. For example, a patient may return to the dental office with a fracture edge of the composite restoration placed the day before. To simulate this immediate repair, in the present study, experimental units received rebonding procedures 24 hours after specimen preparation. It has been stated that the greatest monomer functional groups' radical activity can be found on the composite surface during the first 24 hours after polymerization.[14,27] In addition, the Bis-GMA monomer seems to have the lowest degree of conversion compared with other dimethacrylates used in dentistry.[28] Therefore, Bis-GMA–based composites could have more unreacted double carbon bonds, which might result in a better repair bond strength.[20] The findings in the present study suggest that the availability of carbon double bonds in the surface of the existing polymer might have allowed the chemical bonding between composite materials.[21,27] Thus, it is hypothesized that the chemical bonding between resin-based materials surpassed the effect of surface treatment.

The use of an intermediary bonding agent has an important role in the composite repair. The ability of monomers and solvent systems to penetrate into the composite surface depends on the chemical affinity of materials and the degree of hydration of the composites.[20,29] Most composites are hydrophobic in nature but contain some absorbed water that might improve surface penetration by hydrophilic bonding systems such as the self-etching systems.[20] The effectiveness of bonding agents is improved by their low viscosity, which produces a small contact angle and good wetting properties.[9,27]

Two-step self-etching systems have been designed to simplify bonding procedures by eliminating the separate acid-etching step.[22] In the present study, only groups air-abraded and bonded with the self-etching system produced a repair bond

strength similar to the UTS of the composite. A previous study[20] reported high bond strength when aged restorations were repaired with a self-etching system. These authors also stated that the technique used to apply the self-etching system might influence the rebonding procedure positively and the light brushing motion may allow an easier penetration of the solvent and monomer into the surface to be repaired. The self-etching system Clearfil SE Bond, evaluated in this study, uses an active application as well. Therefore, the high repair bond strength found might be justified by its capacity to wet the air-abraded surface.

The self-etching system Clearfil SE Bond contains the proprietary acid phosphate monomer 10-methacryloyloxydecyl dihydrogen phosphate. A previous study suggested that the specific molecular nature of this functional monomer determines an efficient and stable bond to tooth structure.[30] Based on the high repair bond strengths observed when the self-etching system was used, it can be hypothesized that the acidic monomer might also have a role in the higher capacity to wet the composite surface. Nevertheless, this issue requires further investigation.

The phosphoric acid cleansing of the surface to be repaired demonstrated no significant influence on the bond strength of the self-etching system. Similarly, previous studies showed no increase in bond strength when repaired composites were treated with 37% phosphoric acid.[12,21] It can be suggested that the mild acidic primer of the self-etching system was able to promote an adequate surface cleansing. Thus, the previous acid conditioning seems to be an irrelevant procedure when self-etching systems are used for composite repairs.

The failure modes type 1 (adhesive failure in the composite–repair interface) and type 2 (mixed failures) were the most frequently observed. If a composite repair tends to fracture cohesively (ie, not in the interface), one can assume that the approach selected was appropriate to bear the occlusal loads.[3] In the present study, no predominately cohesive composite failures were observed, irrespective of the high bond strength values found in repaired groups.

The null hypotheses tested in the present study were rejected. When repair procedure was performed 24 hours after composite polymerization, different combinations of surface treatments and bonding procedures presented similar repair bond strengths. However, repair bond strengths similar to the composite UTS could not be reached with only one repair approach (surface treatment or bonding procedure). Moreover, not all repaired groups presented bond strength values comparable to the UTS of the composite.

It should be reemphasized that the aim of the present study was to repair "newly placed restorations" rather than aged ones. Repair approaches tested indicated a good performance of immediate repairs. This security appears to be related to the possible chemical interaction between adhesive materials. Thus, the choice of a repair approach concerning newly placed restorations should be made according to the equipment and materials available in the dental setting.

CONCLUSION

Within the limitations of this study, it was concluded that when repair is performed 24 hours after composite polymerization, the repair techniques tested in this study resulted in similar bond strength. Higher repair bond strength was observed for specimens air abraded and bonded with the self-etching system, irrespective of the phosphoric acid cleansing.

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