Bond Strength of Composite Resin Restoration Repair: Influence of Silane and Adhesive Systems

Natália C Gutierrez¹, Sabrina E Moecke², Taciana MF Caneppele³, Leticia CCC Perote⁴, Graziela R Batista⁵, Maria FRL Huhtalla⁶, Carlos RG Torres⁷

ABSTRACT

Aim: The aim of this study is to evaluate the effect of silane (Si) application and different adhesive systems on the bond strength of composite resin repair.

Materials and methods: One hundred composite truncated cone-shaped specimens were prepared and submitted to 5,000 thermal cycles to simulate existing restorations. Their top surfaces were airborne particle abraded with aluminum oxide, etched with phosphoric acid, and divided into two groups (n = 50) with or without Si application. Each group was divided into five subgroups (n = 10) according to the adhesive system applied: Solobond Plus Primer and Adhesive (SPA)—two-bottle, Solobond Plus adhesive (SA), Admira Bond (A)—one bottle, Futurabond DC (FDC)—self-etch, and Futurabond M (FM)—self-etch. New composite resin was applied over the bonded area. A control group was prepared to evaluate the cohesive strength of the composite resin. Specimens were submitted to tensile stress. Data were analyzed with two-way analysis of variance (ANOVA), and the Tukey and Dunnett tests.

Results: Si application reduced the bond strength of all adhesives (p = 0.001). Groups SA and SPA showed higher bond strengths in relation to other groups (p = 0.01). Groups FDC + Si, FM, SA, and A + Si showed smaller mean bond strength values than that of the control group (p < 0.05).

Conclusion: Previous Si application reduced bond strength values. The two-bottle adhesive showed better results than one-bottle or self-etching systems for composite resin repairs.

Clinical significance: The kind of adhesive system applied for composite resin repairs has a great influence on bond strength values. The use of Si in this situation is not recommended.

Keywords: Adhesive system, Composite resin repair, Laboratory research, Tensile bond strength.

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INTRODUCTION

Composite resins are widely used in dentistry because of the great demand for aesthetic restorations.¹,² Despite all improvements in their formulation, restorations frequently fail because of small or large fractures and require repair or total replacement.³ Total replacement of the defective restoration may represent overtreatment, since parts of the restoration can be clinically and radiographically free of failure and its total removal can increase the cavity size.⁴,⁵

Repair is a more conservative alternative and should be the first option, reducing tooth structure loss and risk of pulpal exposure. Furthermore, repair is also a more rapid and less expensive treatment.⁶,⁷ Adding a new composite resin to an existing one is a well-established clinical procedure,⁸ but some issues still need to be considered.

It may be difficult for the clinician to identify the brand and characteristics of the existing composite resin, which will be exposed to the wet oral environment for some time.⁹ Aged restorations, absorbed water, and other substances from bacterial metabolism and the patient’s diet, such as acids, ethanol, and oils, can lead to matrix softening and degradation.¹⁰–¹² Unlike adding a composite resin layer during the placement of a new restoration, the absence of an oxygen-inhibited layer will hamper the bonding between the old and new material.¹³

To improve bonding between the old and new composite resins, surface roughening with a rotary instrument or airborne-particle abrasion and/or coating of the aged composite resin with bonding agents is required.¹⁴,¹⁵ Si primer application has also been proposed, aiming to promote chemical bonding between the inorganic filler particles that are cut and exposed during the surface roughening procedure.¹⁶,¹⁷ However, this is an additional clinical step and its efficacy is unclear.

The use of an intermediate layer of a hydrophobic fluid resin, such as the adhesive component of a two-bottle fourth-generation bonding system, can promote a chemical bond to the organic matrix or to the silanized filler particles, in addition to creating micromechanical retention by microscopically interlocking into surface irregularities.¹⁸ However, the existence of different adhesive formulations and the increased use of self-etching adhesives can complicate this procedure. Self-etching adhesives

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have acidic and hydrophilic monomers that can compromise the interaction between polymerized composite resin and the new material.\textsuperscript{13}

Although this subject has been studied, disagreement and doubts still exist as to the best technique and adhesive for repairing aged composite resin restorations.\textsuperscript{19} Therefore, the aim of this study is to evaluate the effect of Si application and different adhesive systems on the composite resin repair bonds of a nanohybrid composite resin. The null hypotheses tested were that prior Si application would have no effect on repair bonding and that different adhesive systems would have the same effect on bond strength.

**Materials and Methods**

**Experimental Design**

This study tested two experimental factors: adhesive system (Solobond Adhesive, SA; Solobond Primer + Adhesive, SAP; Admira Bond, A; Futurabond M, FM; and Futurabond DC, FDC) and Si application (with and without). The dependent variable was the tensile bond strength.

**Specimen Preparation**

One hundred truncated cone-shaped specimens (bottom—4 mm; top—2 mm; height—4 mm) were prepared from a nanohybrid composite resin (GrandioSO, Voco, Cuxhaven, Germany), shade A4, to simulate the existing restoration to be repaired. They were built with 2 mm increments of material applied inside a silicone matrix and light polymerized using an LED device (Elipar Free Light 2, 3M ESPE, St. Paul, MN, USA) for 20 seconds with an irradiance of 1,000 mW/cm\(^2\) (Fig. 1).

To simulate an existing restoration on which the repair would be performed, specimens were submitted to artificial aging using a thermocycling machine (ER 3700, ERIOS, Sao Paulo, Brazil). Five thousand cycles were performed with a 5–55 °C temperature change and 30 seconds dwell time. Subsequently, the tops of the specimens were airborne particle abraded with aluminum oxide, cleaned with air/water spray, etched with phosphoric acid for 15 seconds, washed, and dried with compressed air.

A control group was prepared to evaluate the cohesive tensile strength of nonrepaired material. For that, a double-truncated silicone cone-shaped matrix was used, and the same composite resin was applied without any bonding interface, which was also thermal cycled (Fig. 2).

**Repair Process**

The specimens were divided into two groups (\(n = 50\)) with or without the application of a Si coupling agent (Ceramic Bond, Voco). Si was applied for 60 seconds followed by a jet of compressed air. Each group was divided into five subgroups (\(n = 10\)) according to the adhesive system applied (Fig. 3A).

- **SA**—the adhesive component of the two-bottle system was applied, followed by air thinning and light polymerizing for 10 seconds.
- **SPA**—the primer component of the two-bottle system was applied, followed by air thinning. Adhesive was then applied, followed by air thinning and light polymerizing for 10 seconds.
- **A**—the one-bottle system was applied, followed by air thinning and light polymerizing for 10 seconds.
- **FDC**—the self-etching dual-cure adhesive was applied for 20 seconds with constant agitation, followed by air thinning and light polymerizing for 10 seconds.
- **FM**—the self-etching adhesive was applied for 20 seconds with constant agitation, followed by air thinning and light polymerizing for 10 seconds.

A double-truncated silicone cone-shaped matrix was used to hold the aged specimen, and the same composite resin was applied (GrandioSO, Voco; shade A1) to simulate repair over an existing restoration (Figs 3B and C). This procedure created a double-truncated cone-shaped specimen with aged and new material bonded at the interface (Fig. 3D). Table 1 shows all materials used in this study, their manufacturers, and their components.

Figs 1A to D: Preparation of single-truncated cone-shaped specimen. (A) Cross section of the silicone matrix; (B) Upper view of the matrix; (C) Incremental application of the composite shade A4; (D) Specimen prepared for simulation of an old restoration
All specimens were attached to a holder (Figs 4A and B) and submitted to tensile stress in a universal testing machine DL-200 MF (Emic, Sao Jose dos Pinhais, Brazil) with a crosshead speed of 1 mm/s (Figs 4C and D).

Statistical Analysis
Data normality was checked using the Kolmogorov–Smirnov test. The Dunnett test was performed to compare the data of different groups in relation to the control. The results of different treatments were compared using two-way ANOVA and the Tukey tests. Statistical analyses were performed with the software Statistics for Windows (Statsoft, Tulsa, OK, USA) with a significance level of 5%.

Results
The bond strength values for all experimental conditions and the results of the Dunnett test are shown in Figure 5. Groups bonded
## Table 1: Materials, their manufacturer, and their components

<table>
<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
<th>Components</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>GrandioSO</td>
<td>Voco</td>
<td>Matrix resin: Bisphenol A-glycidyl methacrylate (Bis-GMA), bisphenol A-ethoxylated dimethacrylate (Bis-EMA), triethylene glycol dimethacrylate (TEGDMA), camphorquinone (CQ), Amina, dibutylhydroxytoluene (BHT) Inorganic content: nanoparticles SiO$_2$: 20–40 nm; glass ceramic: 1 μm Filler content: 89% weight, 73% vol</td>
<td>1.3</td>
</tr>
<tr>
<td>Solobond Plus</td>
<td>Voco</td>
<td>Primer—2-Hydroxyethyl methacrylate (HEMA), hydroxypropyl methacrylate, acetone</td>
<td>1.3</td>
</tr>
<tr>
<td>Admira bond</td>
<td>Voco</td>
<td>Acetone, Ormocer, Bis-GMA, HEMA, BHT, and acidic adhesive monomer</td>
<td>2.0</td>
</tr>
<tr>
<td>FM</td>
<td>Voco</td>
<td>HEMA, Bis-GMA, ethanol, acidic adhesive monomer, UDMA, Camphorquinone</td>
<td>1.4</td>
</tr>
<tr>
<td>FDC</td>
<td>Voco</td>
<td>Bis-GMA, HEMA, TMPTMA, methacryl phosphoric acid ester, camphorquinone, BHT, and ethanol</td>
<td>1.4</td>
</tr>
<tr>
<td>Ceramic bond</td>
<td>Voco</td>
<td>Acetone, 3-methacryl oxypropyl trimethoxy silane, and isopropanol</td>
<td>4.5</td>
</tr>
</tbody>
</table>

Cuxhaven, Germany

Figs 4A to D: Tensile bond strength test. (A) First cone of the double specimen inside the holder; (B) Holder closure on second cone; (C) Specimen during the test; (D) Specimen after test

Fig. 5: Mean tensile bond strength (MPa) and standard deviation (±SD). Groups: SA, SPA, Admira bond (A), FDC, and FM. *Significant differences in relation to the control group ($p < 0.05$)
with Admira and DC with Si and groups DC with and without Si showed smaller means than the cohesive values in the control group.

The results of two-way ANOVA showed significant differences for the two factors: Si application (p = 0.0012) and adhesive system (p = 0.001). The Tukey test showed that the Si application resulted in smaller means (17.36 ± 6.63 MPa) than when not applied (21.05 ± 6.93 MPa). The results of the Tukey test for the kind of adhesive are shown in Table 2. The Solobond system, with or without primer application, presented significantly higher means than those of the other systems tested.

**Discussion**

Composite resin restorations are generally repaired years after the original placement. During the aging process, different phenomena occur, including water sorption and hydrolytic, thermal, and chemical degradation, which can negatively affect the success of a composite resin repair. An aging procedure is necessary to simulate clinical conditions when composite resin repairs are tested *in vitro*. Thermocycling, subjecting specimens to extreme temperatures, is a laboratory method for simulating oral conditions. According to ISO standards, 500 thermocycles in water temperatures between 5 °C and 55 °C are considered an appropriate test for aging dental materials. However, according to Ozcan et al., 5,000 thermocycles were the most effective aging method when the bond strength of composite resin to composite resin was tested; therefore, this number was applied in our study to simulate an existing restoration.

The surface treatment on an existing composite resin is intended to remove the superficial layer altered by saliva and increase the surface area available for bonding by creating surface irregularities. In the present study, the surfaces to be bonded were airborne particle abraded with aluminum oxide. Previous studies concluded that this pretreatment provided the highest composite resin repair bond strength compared with roughening with different grit diamond rotary instruments.

Both the null hypotheses tested in this study were rejected because prior Si application and the kind of adhesive system applied had a significant effect on bond strength. Si application is recommended for bonding indirect glass–ceramic restorations to the tooth structure, as well as for ceramic repair. Glass–ceramic materials are not able to bond chemically to adhesive methacrylate monomers without a Si coupling agent to promote the interaction between the organic monomers and inorganic components. However, when bonding indirect composite resin restorations, as well as when a repair of direct or indirect composite resin is indicated, the material surface contains a methacrylate polymer network, which can chemically interact directly with the bonding system. However, as the main components of the material are inorganic glass fillers exposed by airborne-particle abrasion or grinding with a diamond rotary instrument, it will not bond chemically to the adhesive applied. As a result, Si could play an important role in the bonding process by creating a covalent chemical bond between the glass particles and monomers in an adhesive system.

In this study, the use of Si decreased the bond strength of the repair to the aged composite resin. Although Si interacts with inorganic fillers, it forms a thick and multiphase interfacial layer between the composite resin and the adhesive, impairing the direct interaction of the methacrylate monomers from the adhesive with the polymer network of the already polymerized composite resin. Therefore, our study is consistent with a previous report that suggested Si application should not be performed. Cho et al. reported that silanization did not improve repair bond strength. Papacchini et al. reported that Si application did not physically or chemically improve repair bond strength.

The two-bottle adhesive system Solobond Plus, with or without primer application, showed significantly higher bond strength values than those of FM, FDC, or Admira. The adhesive component of the Solobond system is more viscous than that of other adhesives tested, creating a thicker layer, with the underlying surface already completely polymerized before the new composite resin is applied, even with the presence of an oxygen inhibiting layer. Furthermore, the adhesive bottle used as a final layer has in its formulation highly hydrophobic molecules, such as Bis-GMA, which can result in an improved interaction with the composite resin polymer of the aged material.

Another explanation is that the Solobond Plus system does not contain the acidic functional monomers available in all the self-etching adhesives tested. Materials with a higher concentration of hydrophobic monomers than hydrophilic and acidic monomers usually have a better degree of conversion. Some studies have reported that the low pH of some adhesives can sometimes interfere with polymerization because of the interaction with a tertiary amine in the composition of most light-polymerized materials. The manufacturer of the Admira bond system, although not considered a self-etching adhesive, recommends a total acid etching before application over tooth structure. However, the adhesive contains acidic monomers and showed similar bond strength values as the self-etching ones. According to Sanares et al., the bond strength of acidic adhesives is compromised because their acidic characteristic results in a lower degree of polymerization. The authors also suggested that this inhibition layer with unpolymerized acidic and hydrophilic monomers can compromise the new composite resin polymerization process. The deficient polymerization of the adhesive layer can also increase water sorption and polymer network degradation. Self-etching adhesives contain more hydrophilic monomers and solvents, which may not be completely removed by the air thinning step, impairing the polymer network formation. Therefore, the drying procedure should be carefully performed to improve solvent evaporation.

Primer application did not improve the bond strength of the two-bottle system (Fig. 5 and Table 2). The primer component in the two-bottle adhesive system is mainly composed of hydrophilic monomers and solvents designed to impregnate the wet dentin collagen network exposed by acid etching. However, the surface can be air dried when it is bonded to an existing composite resin.
and there is no need for a special hydrophilic monomer. Therefore, the primer step should be omitted. These findings are consistent with those of previous studies. In comparison with the control group testing the cohesive strength of the composite resin, Si application and self-etching adhesives resulted in significantly lower bond strength values (Table I). According to our results, these systems should be avoided when composite resin restorations are repaired.

Since this was an in vitro study, clinical extrapolation should be carefully done. Further investigations should focus on in vitro evaluation with different materials and aging protocols, besides clinical evaluation of composite resin restoration repair.

**CONCLUSION**

Within the limitations of this in vitro study, it was concluded that Si application before the repair of a nanohybrid composite resin reduced the bond strength values. The two-bottle adhesive tested showed better results for composite resin repair than the one-bottle or self-etching systems, probably due to different monomer compositions, which influences the degree of polymerization and interaction with the aged composite. The use of Si and self-etching adhesives should be avoided when composite resin restorations are repaired.

**CLINICAL SIGNIFICANCE**

The kind of adhesive system applied for composite resin repairs has a great influence on bond strength values. The use of Si in this situation is not recommended.

**REFERENCES**