

Bond strength of self-adhesive resin cements to composite submitted to different surface pretreatments

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Objectives: Extensively destroyed teeth are commonly restored with composite resin before cavity preparation for indirect restorations. The longevity of the restoration can be related to the proper bonding of the resin cement to the composite. This study aimed to evaluate the microshear bond strength of two self-adhesive resin cements to composite resin. **Material and Methods:** Composite discs were subject to one of six different surface pretreatments: none (control), 35% phosphoric acid etching for 30 seconds (PA), application of silane (silane), PA + silane, PA + adhesive, or PA + silane + adhesive ($n = 6$). A silicone mold containing a cylindrical orifice (1 mm² diameter) was placed over the composite resin. RelyX Unicem (3M ESPE) or BisCem (Bisco Inc.) self-adhesive resin cement was inserted into the orifices and light-cured. Self-adhesive cement cylinders were submitted to shear loading. Data were analyzed by two-way ANOVA and Tukey's test ($p < 0.05$). **Results:** Independent of the cement used, the PA + Silane + Adhesive group showed higher microshear bond strength than those of the PA and PA + Silane groups. There was no difference among the other treatments. Unicem presented higher bond strength than BisCem for all experimental conditions. **Conclusions:** Pretreatments of the composite resin surface might have an effect on the bond strength of self-adhesive resin cements to this substrate. (*Restor Dent Endod* 2014;39(1):12-16)

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Introduction

Improvements in adhesive luting systems and ceramic materials have resulted in more conservative tooth preparation procedures for the retention of indirect restorations. Conservative tooth preparations may require luting materials with improved adhesive ability for the retention of restorations.^{1,2} For many years, adhesive systems have been applied with resin-based luting agents to lute metal-free indirect restorations. However, multistep luting protocols increase the technique sensitivity and clinical time.³⁻⁷ Simplified luting agents are gaining increased popularity in this scenario.

Self-adhesive resin cements (SARCs) have been marketed to simplify the clinical procedures and overcome the technique sensitivity of multistep luting systems. According to manufacturers, SARCs do not require pretreatment and their application is accomplished using a single clinical step. Several studies have evaluated the bonding ability of SARCs to dental tissues, with results generally depending on the materials tested.⁸⁻¹¹ However, the presence of extensively destroyed coronal structures

is relatively common in the clinical setting, requiring the preparation of a composite restoration (core) to serve as an abutment. Fiber posts can be required to retain the core, which is usually built-up using composite resin.¹² Therefore, the retention of indirect restorations is dependent on the bonding of the resin cement to the composite core.

Several surface pretreatment protocols have been evaluated to improve the bond strength of repairing composite resin to old one after acid-etching and silane application.¹³⁻¹⁶ The results of these previous studies indicate that composite pretreatments might improve the repair bond strengths. Treating the composite surface before luting procedures could also enhance the bonding of SARC to composite cores. However, few studies have evaluated the bonding of SARC to composite resins. Thus, the aim of this study was to evaluate the effect of different surface treatments of composite resin on the bond strength of SARC to this substrate. The hypothesis tested was that treating the composite surface would improve the bonding potential of the SARC.

Materials and Methods

This *in vitro* study involved a 2 × 6 factorial design (*n* = 6 per group). The factors under evaluation were: resin cement (2 levels) and composite pre-treatment (6 levels). The SARC tested were RelyX Unicem clicker (3M ESPE, St. Paul, MN, USA) and BisCem (Bisco INC., Schaumburg, IL, USA). Table 1 depicts the luting agent compositions. The SARC were bonded to composite resin discs (2 mm height, 6 mm diameter) of Tetric Ceram (shade B3, Ivoclar Vivadent, Schaan, Liechtenstein). The response variables were microshear bond strength (MPa) to composite and failure mode.

A total of 72 composite resin discs was prepared by inserting the material into a metallic mold, which was covered with a polyester strip. The composite was inserted using a bulk increment and light-cured for 45 s using a light-emitting diode unit (Radii Cal, SDI, Bayswater, Victoria, Australia) with 800-mW/cm² irradiance. After light-curing, the discs were removed and embedded in acrylic resin (Jet, Clássico, São Paulo, SP, Brazil), leaving

one of the surfaces exposed. After storage in water at 37°C for 7 days in an incubator (Marconi, Piracicaba, SP, Brazil), the exposed composite resin surface received one of following pretreatments (12 discs per treatment):

- Control: No surface treatment was performed.
- Phosphoric acid (PA): The surface was etched with 35% PA (3M ESPE) for 15 seconds, rinsed with water for 10 seconds, and air-dried using compressed air for 10 seconds.
- Silane: A layer of a silane-coupling agent (RelyX Ceramic Primer, 3M ESPE) was applied and left undisturbed for 60 seconds, and then gently air-dried for 10 seconds.
- PA + Silane: 35% PA was applied, and the silane agent was applied after the procedures described for the PA group.
- PA + Adhesive: The non-solvated unfilled resin of the Scotchbond Multipurpose Plus system (3M ESPE,) was applied after acid-etching.
- PA + Silane + Adhesive: The composite surface was acid-etched, cleaned, and silanized before application of the unfilled resin.

To obtain resin cement cylinders for shear testing, elastomer molds with internal cylindrical orifices (1 mm diameter, 2 mm height) were placed over the prepared surfaces of the composite resin. The unfilled resin was photoactivated for 20 seconds (when applicable) only after positioning of the elastomer molds. The two pastes of each SARC were mixed for 10 seconds, and the mixed cement was inserted into the mold using a dental explorer. Light activation was performed for 20 seconds, the molds were removed, and the specimens were stored in 100% relative humidity. Six composite discs were used for each resin cement-composite pretreatment condition.

After 24 hours, a microshear bond test was conducted on a mechanical testing machine (Instron 5940, Instron, Canton, MA, USA). A thin steel wire (0.2 mm diameter) was looped around each cylinder and aligned with the bonded interface. A shear load was applied to the base of the cylinder at a crosshead speed of 0.5 mm/min until failure. The average value of three bonded cylinders for each composite disc was recorded as the microshear bond strength (MPa) for each composite disc. Data passed the

Table 1. Composition of the self-adhesive resin cements tested

Material	Mains components*
BisCem	Base: Bisphenol-A glycidyl dimethacrylate, uncured dimethacrylate monomer, glass filler. Catalyst: Phosphate acidic monomer, glass fillers.
Unicem	Base: Methacrylate monomers containing acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizer. Catalyst: Methacrylate monomer, alkaline fillers, silanated fillers, initiator components.

*As provided by the manufacturers.

normality ($p = 0.097$) and equal variance ($p = 0.067$) tests, and therefore were statistically analyzed using two-way ANOVA (resin cement \times composite pretreatment) and Tukey's *post hoc* test ($p < 0.05$) using the software SigmaStat 3.5 (Systat Software, Erkrath, Germany).

Results

Results for bond strength are shown in Table 2. The factors "resin cement" ($p < 0.001$) and "composite pretreatment" ($p = 0.009$) were both significant, whereas the interaction between the two factors was not significant ($p = 0.105$). The PA + Silane + Adhesive group showed significantly higher microshear bond strength than the PA and PA + Silane groups. There were no differences among the other surface pretreatments. Unicem showed a higher microshear bond strength than BisCem ($p < 0.001$), irrespective of the composite pretreatment used. A predominance (above 90%) of adhesive failures was observed regardless of the surface treatment or cement tested.

Discussion

One of the main bonding mechanisms of SARC to dental tissues is related to the reaction between the acidic monomers of the cement with calcium present in the dentin and enamel, as SARC present limited ability to produce an effective hybrid layer.¹⁷⁻²⁰ However, the chemical reaction is limited when SARC are bonded to the composite resin used as a core. Additionally, the chemical reaction between SARC and dental calcium aids in buffering the pH of the cement and improves its mechanical properties.^{21,22} Theoretically, chemical bonding between methacrylate monomers is the main bonding mechanism between SARC and composite resin. However, there is a reduced amount of unreacted monomers on the surface of cured composite resin to react with the cement.^{13,14} In the present study, no

pretreatment of the composite surface was able to improve the bond strength of the SARC, although there were differences among the pretreatments evaluated. The tested hypothesis was, therefore, rejected.

An important observation of this study was that the effect of the pretreatments was similar for both SARC evaluated. Surface etching with PA is commonly used in repair procedures of composite resin restorations, although in some studies, etching with PA was unable to promote a significant increase in microretention on the composite surface.^{14,15} Acid etching primarily serves to clean the composite surface, resulting in improved surface energy.¹⁶ Despite the possibly increased wetting of the SARC on the cleaned composite surface, etching with PA did not provide higher bonding potential compared to the control.

SARC present an acidic character in the initial moments after mixing.¹⁸ This low pH is important for proper etching of the dental tissues.¹⁷ Reaction of the acidic methacrylates with the alkaline ions leached from the acid-soluble glass particles present in SARC also aids the materials in becoming more hydrophobic with time.¹⁸ The pH-buffering effect is important to permit adequate free-radical polymerization of the SARC and to improve their mechanical stability. Acid-etched surfaces may present residual acidity, which could impair both pH buffering and cement polymerization. Thus, a possible benefit of better wetting after acid etching could be counterbalanced by the reduced mechanical properties of the cement.^{21,23}

Another surface pretreatment evaluated in this study was the use of silane-coupling agent. Silanes promote chemical bonding between inorganic surfaces and polymeric molecules.²⁴ Inorganic fillers in the composite resin are bonded to the organic matrix monomers by silanes, and exposure of the fillers would allow chemical coupling with the glass particles. In the present study, the use of silane was unable to improve the bonding of either SARC to the composite resin. The low availability of exposed glass particles on the composite surface was most likely

Table 2. Means (standard deviations) for microshear bond strength, MPa

Pre-treatment	Resin Cement	
	Unicem	BisCem
Control	23.9 \pm 8.3 ^{Aab}	12.4 \pm 3.0 ^{Bab}
PA	19.6 \pm 5.0 ^{Ab}	14.1 \pm 5.3 ^{Bb}
PA + Silane	17.8 \pm 4.4 ^{Ab}	13.7 \pm 3.6 ^{Bb}
PA + Silane + Adhesive	31.2 \pm 11.9 ^{Aa}	19.5 \pm 4.2 ^{Ba}
PA + Adhesive	30.3 \pm 9.1 ^{Aab}	14.1 \pm 2.5 ^{Bab}
Silane	26.9 \pm 2.4 ^{Aab}	14.2 \pm 5.8 ^{Bab}

Distinct lowercase letters in the same column indicate differences between pre-treatments; distinct uppercase letters in the same line indicate differences between resin cements ($p < 0.05$).

responsible for those findings. The use of silane after acid-etching also did not alter the bond strength compared to the control group. It has been demonstrated that etching with PA might enhance the reactivity between silica surfaces and silane-coupling agents.^{25,26} The absence of a significant effect in the present study demonstrates that the acid-etching protocol used was unable to expose the glass particles on the composite surface effectively, thereby impairing the chemical coupling through silanization.

The group with adhesive application after acid etching and silanization showed the highest bond strength values, with significant differences compared with use of PA alone or PA + silane. In contrast, application of adhesive alone did not show significant differences from the other treatments. A possible explanation is that the combined use of silane and adhesive improved the wetting of the adhesive on the composite resin, whereas a possible negative effect of residual acidity did not affect the polymerization of the adhesive. Irrespective of the surface pretreatment performed, Unicem showed higher bond strength than BisCem. Other studies reported similar results when bonding SARC to tooth tissues and ceramics.^{10,11,26} Unicem has a higher pH-neutralization ability than BisCem, improving its mechanical properties.^{21,22}

The present results indicate that different pretreatments of the composite resin surface may have distinct effects on the bonding potential of SARC. However, no pretreatment evaluated was able to improve the bond strength to composite resin as compared with the control surfaces. Other surface treatments used to improve the bonding to cured composite surfaces should be evaluated in future studies, such as the use of alumina sandblasting or etching with hydrofluoric acid.

Conclusions

Within the limitations of the present study, surface treatments might have an effect on the bonding of SARC to composites.

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