Effect of Silanization on Microtensile Bond Strength of Different Resin Cements to a Lithium Disilicate Glass Ceramic

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ABSTRACT

Aim: This study evaluated the influence of a silane-coupling agent on the bond strength of a self-adhesive cement and a conventional resin cement to a lithium disilicate glass ceramic.

Materials and methods: A total of eight ceramic blocks were fabricated and divided into four groups (n = 2). In groups 1 and 3, ceramic surfaces were etched with hydrofluoric acid 10% for 20 seconds, rinsed for 30 seconds, and air-dried. One layer of a silane agent was applied onto all ceramic specimens and air-dried for 30 seconds. In groups 2 and 4, ceramic surfaces were etched with hydrofluoric acid, rinsed, and air-dried without application of the silane-coupling agent. The ceramic blocks were bonded to a block of composite with a self-adhesive resin cement or with a conventional resin cement, according to the manufacturer’s instructions. After 24 hours in distilled water at 37°C, the specimens were sectioned perpendicular to the bonding interface area to obtain beams with a bonding area of 0.8 mm² and submitted to a microtensile bond strength test at a crosshead speed of 0.5 mm/min. Data were statistically analyzed with one-way analysis of variance and the Games–Howell post hoc test (p = 0.05). Fractured specimens were examined under optical microscopy at 40× magnification.

Results: Silanization resulted in higher microtensile bond strength compared to groups without silane. No significant differences were found between the conventional resin cement and the self-adhesive resin cement with silane agent (p = 0.983), and without silane agent (p = 0.877).

Conclusion: Silanization appears to be crucial for resin bonding to a lithium disilicate-based ceramic, regardless of the resin cement used. The self-adhesive resin cement performed as well as the conventional resin cement.

Clinical significance: Applying one layer of a silane-coupling agent after etching the ceramic surface with hydrofluoric acid 10% enhanced the bond strength between resin cements and a glass ceramic.

Keywords: Laboratory research, Microtensile bond strength, Resin cement, Silanization.


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Conflict of interest: None

INTRODUCTION

Success with resin-bonded all-ceramic restorations is highly dependent on obtaining a durable and trustworthy bond, which must integrate all parts of the system into one consistent structure.1 Adhesive bonding depends on the surface energy and wettability of the adherent by the adhesive.2,3 The adhesion between resin-based composites and dental ceramics is the result of a physicochemical interaction across the interface between the adhesive and the substrate.2 A newly introduced lithium disilicate glass ceramic (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) may be adhesively cemented. Bonding to this type of ceramic material is based in a mechanical and chemical interaction, promoted by hydrofluoric acid and the silane-coupling agent respectively.4 Hydrofluoric acid etching removes the glassy matrix and the second crystalline phase, resulting in irregularities in the lithium disilicate crystals of the IPS e.max system, allowing a mechanical union with resin composites.5-7 The silane-coupling
agent presents bifunctional characteristics, promoting a
chemical interaction between the silica in the glass phase
of ceramics and the methacrylate groups of the resin
through siloxane bonds.\textsuperscript{8,9}

Silane agents have been used since 1949 to improve
the bonding among adhesive resins with organic com-
ponents, ceramics, and metals, but in 1977 Eames et al\textsuperscript{10}
suggested the use of silane agent in dentistry. Previous
studies report the effectiveness of the silane-coupling
agent to improve the bond strength between resin com-
posites and ceramics\textsuperscript{11,12} and between resin cements and
ceramics\textsuperscript{9,13,14}; however, some studies\textsuperscript{2,15-17} question the
use of silane. Some authors concluded that the application
of a silane agent did not show a significant effect in
the bond strength between ceramic materials and resin
cements.\textsuperscript{2,15,17}

To simplify the clinical procedures and overcome the
technique sensitivity of multistep luting systems, resin
cements that combine an acid primer and cement in one
application, called self-adhesive resin cements, have been
introduced.\textsuperscript{3,15} Self-adhesive resin cements do not require
pretreatment and their application is accomplished using
a single clinical step. Nevertheless, there are controversies
in the literature about the bonding capacity of this type
of adhesive agent to the ceramic surface treated or not
with a silane-coupling agent.

Therefore, the aim of the present study was to
evaluate the influence of silane-coupling agent on the
microtensile bond strength of a self-adhesive cement and
a conventional resin cement to a glass ceramic reinforced
with lithium disilicate, the IPS e.max system. The null
hypotheses tested were as follows: (1) the silane agent
does not influence the bond strength among resin cements
and the ceramic reinforced with lithium disilicate; (2) the
microtensile bond strength is not associated with the type
of cement used.

### MATERIALS AND METHODS

#### Ceramic Blocks

A total of eight rectangular blocks (11 mm long × 9 mm
wide × 4 mm thick) of IPS e.max Press were fabricated in
accordance with the manufacturer’s instructions.

Rectangular wax patterns were created, sprued, and
attached to a muffle base with a surrounding paper
cylinder. The wax patterns were invested with phosphate-
based material (IPS PressVest Speed, Ivoclar Vivadent),
and the wax was eliminated in an automatic furnace
(Vulcan A-550, Degussa-Ney, Yucaipa, California, USA)
at 850°C for 1 hour. The IPS e.max Press ceramic ingots
were pressed into the molds in an automatic press furnace
(Ep 500, Ivoclar Vivadent). After cooling, the specimens
were divested and submitted to wet polishing with silicon
carborundum grinding paper (grits #220, #360, and #600)
(Acqua Flex-Norton, São Paulo, Brazil) in a polishing machine
(Panambra, São Paulo, Brazil). The blocks were cleaned in
ultrasonic bath with distilled water for 5 minutes, air-dried,
and divided into four groups (n = 2) according to the resin
cement and the surface treatment performed.

#### Composite Resin Blocks

A total of eight rectangular blocks (11 mm long × 9 mm
wide × 4 mm thick) of composite Filtek Z350, shade A2
(3M ESPE, St Paul, Minnesota, USA) were fabricated.
To manufacture the composite resin blocks, molds
from all the ceramic blocks were obtained with an
elastomeric mold (Virtual, Ivoclar Vivadent, Schaan,
Liechtenstein). The composite resin was inserted in the
denture in a polishing machine (Panambra, São Paulo, Brazil).
The blocks were cleaned in an ultrasonic bath with distilled water
for 5 minutes, air-dried, and divided into four groups (n = 2)
according to the resin cement and the surface treatment performed.

<table>
<thead>
<tr>
<th>Product name</th>
<th>Composition</th>
<th>Manufacturer</th>
<th>Batch number</th>
<th>Instructions for use</th>
</tr>
</thead>
<tbody>
<tr>
<td>IPS e.max Pressing ingots</td>
<td>SiO₂; Li₂O; K₂O; MgO; ZnO; Al₂O₃; P₂O₅; other oxides</td>
<td>Ivoclar Vivadent, Schaan, Liechtenstein</td>
<td>R37944</td>
<td>–Apply on the ceramic surface for 60 s –Gently air-dry for 30 s</td>
</tr>
<tr>
<td>Monobond S</td>
<td>3-methacryloxy-propyl-trimethoxysilane (1 wt%), water/ethanol solution containing acetic acid set to pH 4 (99 wt%)</td>
<td>Ivoclar Vivadent, Schaan, Liechtenstein</td>
<td>P70737</td>
<td>–Apply on the surface –Lute resin block using light pressure –Light polymerize for 20 s each side</td>
</tr>
<tr>
<td>SpeedCEM</td>
<td>Acidic monomers, dimethacrylates, barium glass, ytterbium trifluoride, co-polymer, silicon dioxide, catalysts, stabilizers, pigments</td>
<td>Ivoclar Vivadent, Schaan, Liechtenstein</td>
<td>S33619</td>
<td>–Apply on surface –Lute resin block using light pressure –Light polymerize for 20 s each side</td>
</tr>
<tr>
<td>Variolink II</td>
<td>BisGMA, TEGDMA, UDMA, benzoyl peroxide, inorganic fillers, ytterbium trifluoride, Ba-Al fluorosilicate glass, spheroidal mixed oxide, initiator, stabilizers, pigments</td>
<td>Ivoclar Vivadent, Schaan, Liechtenstein</td>
<td>R69347</td>
<td>–Mix base and catalyst paste –Lute resin block using light pressure –Light cure for 40 s from each side</td>
</tr>
</tbody>
</table>

BisGMA: bisphenol A glycidylmethacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate
Ceramic Surface Treatment

The eight ceramic blocks were randomly divided into four groups (n=2). In groups 1 and 3, the test surface of the ceramic blocks was etched with hydrofluoric acid 10% (Condac Porcelana, FGM, Joinville/SC, Brazil) for 20 seconds, rinsed with water for 30 seconds, and air-dried for 30 seconds. One layer of a silane-coupling agent (Monobond S, Ivoclar Vivadent, Schaan, Liechtenstein) was applied onto all ceramic specimens and air-dried for 30 seconds. In groups 2 and 4, the ceramic surfaces were etched with hydrofluoric acid 10% for 20 seconds, rinsed with water for 30 seconds, and air-dried for 30 seconds without application of the silane-coupling agent.

In groups 1 and 2, the self-adhesive resin cement SpeedCEM (Ivoclar Vivadent, Schaan, Liechtenstein) was applied directly to the previously treated ceramic surface. In groups 3 and 4, the resin cement Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein) was applied following the manufacturer’s instructions.

Cementing the Composite to the Ceramic

After the resin cement was applied in the ceramic surface, the composite resin block was positioned over the resin cement under a 500-gm static load for 2 minutes.4 The excess cement was removed with a disposable micro-brush. Light activation was performed for 40 seconds at right angles to each of the IPS e.max Press/Filtek Z350 margins using an LED source (Translux Power Blue, Heraeus Kulzer, Germany) with a final 40-second light exposure from the top surface.

Microtensile Bond Strength Testing

After 24 hours of storage in distilled water at 37°C, the specimens were sectioned perpendicular to the bonding interface area to obtain beams with a bonding area of 0.8 mm² using a water-cooled diamond blade (Buehler Wafering Blades, Buehler Ltd., Illinois, USA) in a low-speed saw machine (Isomet 1000, Buehler, Lake Bluff, Illinois, USA). The cross-sectional area of the bond interface of each beam was measured using a digital caliper (KingTools, São Paulo, Brazil). Thirty specimens from each group were randomly selected for the microtensile bond strength test.

Each beam was fixed to the grips of a microtensile device using a cyanoacrylate adhesive, and the microtensile bond test was conducted in a testing machine (Instron 4444, Instron Corp., Canton, Massachusetts, USA) at 0.5 mm/min crosshead speed until failure.

The fractured specimens were observed under optical microscopy (Olympus Corp., Tokyo, Japan) at 40× magnification.

Table 2: Descriptive statistics (in MPa) of microtensile bond strengths of the four groups tested

<table>
<thead>
<tr>
<th>Groups</th>
<th>Adhesive</th>
<th>Silane</th>
<th>Mean</th>
<th>SD</th>
<th>Min</th>
<th>Max</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>SpeedCEM</td>
<td>Yes</td>
<td>22.162a</td>
<td>10.32</td>
<td>5.75</td>
<td>42.87</td>
</tr>
<tr>
<td>2</td>
<td>SpeedCEM</td>
<td>No</td>
<td>14.434b</td>
<td>5.08</td>
<td>6.02</td>
<td>23.98</td>
</tr>
<tr>
<td>3</td>
<td>Variolink</td>
<td>Yes</td>
<td>21.485a</td>
<td>4.66</td>
<td>7.78</td>
<td>27.89</td>
</tr>
<tr>
<td>4</td>
<td>Variolink</td>
<td>No</td>
<td>15.819b</td>
<td>4.08</td>
<td>5.79</td>
<td>23.14</td>
</tr>
</tbody>
</table>

Means with the same letters are not significantly different by Games–Howell post hoc test at 5%; SD: standard deviation

Failure modes were classified as adhesive, mixed, or cohesive. In the event of spontaneous debonding, specimens were excluded from the statistical analysis. Bond strength data were statistically analyzed with one-way analysis of variance (ANOVA) and the Games–Howell post hoc test (p=0.05).

RESULTS

Descriptive statistics of the microtensile bond strength (MPa) of the different subgroups are illustrated in Table 2. Normality of the distribution was attested with Shapiro–Wilks test (p > 0.05). One-way ANOVA showed the presence of significant differences among the groups (p < 0.0001). Games–Howell post hoc test showed that silane coupling resulted in higher microtensile bond strength compared to groups without silane. No significant differences were found between Variolink and SpeedCEM with silane coupling (p=0.983) and between Variolink and SpeedCEM without silane coupling (p=0.877).

DISCUSSION

The results of the present study revealed that the treatment of the glass-ceramic surface with the silane-coupling agent increased the microtensile bond strength for both evaluated resin cements; thus, the first null hypothesis must be rejected.

The type of cement did not influence the bond strength values. The self-adhesive and the conventional resin-based cements presented similar performance; therefore, the second hypothesis must be accepted.

Applying silane-coupling agent on the ceramic surface after etching with hydrofluoric acid is a well-established protocol.11,18 Due to its bifunctional characteristics, application of silane on the etched ceramic surface may increase the chemical bonding between the ceramic and resin materials once the silane agents, by means of siloxanic bonds, couple the silica oxides present in ceramics to the organic matrix of resin cements.8

Our results reveal that application of silane was fundamental in order to achieve durable adhesion between both resin cements tested and a glass ceramic.
reinforced with lithium disilicate, suggesting that beyond the micromechanical bond promoted by the etching with hydrofluoric acid, the siloxanic bond obtained with the silane-coupling agent application was important.\textsuperscript{19} These results are in agreement with other studies\textsuperscript{9,13,14} that reported the effectiveness of the silane-coupling agent in improving the bond strength of ceramic materials and resin cements.

SpeedCem is a self-adhesive resin cement with adhesive monomers with long methacrylate chains and a phosphoric acid group which can establish chemical bonds with the dental structure and with ceramic surfaces, so no additional adhesive agents are needed.\textsuperscript{20} In the present study, when the silane-coupling agent was applied prior to the application of the self-adhesive cement, statistically higher bond strength was obtained. This result could be explained by the fact that the surface treatment of the ceramic surface with the silane-coupling agent promotes a chemical bond between the ceramic and the resin cement and increases the cement wettability of the ceramic surface, improving the contact of the resin cement with the ceramic surface and enhancing the infiltration of the resin cement in the irregularities obtained with the acid etching.\textsuperscript{21}

The conventional resin cement Variolink II is an etch-and-rinse, dual-curing luting composite resin with the presence of bisphenol A glycidylmethacrylate (BisGMA), urethane dimethacrylate, triethylene glycol dimethacrylate (TEGDMA), and filler particles. The treatment of the ceramic surface with the silane-coupling agent previously to the resin cement application provides a better wettability of the ceramic surface and also a chemical bond between the reactive organic groups of the silane-coupling agent and the resin molecules of the resin cement, like BisGMA and TEGDMA.\textsuperscript{5} In addition, the hydrolyzable monovalent groups, present in the silane-coupling agent composition, chemically bond the silica present in the glass ceramic.\textsuperscript{7}

Unlike other studies that questioned the use of silane-coupling agents in the etched ceramic surface,\textsuperscript{2,15,16,22} in this study, the application of the silane-coupling agent was fundamental for the union of resin cements with the ceramic.

Monobond S ceramic primer is a single-phase preactivated solution based on the 3-methacryloxypropyltrimethoxysilane molecules. These solutions are composed of bifunctional molecules that bond silicon dioxide with the \(-\text{OH}\) groups on the ceramic surface,\textsuperscript{23} and normally contain a silane coupler and a weak acid, which enhances the formation of siloxane bonds.\textsuperscript{24} They also have a degradable functional group that copolymerizes with the organic matrix of the resin.\textsuperscript{25}

Various methods are available for assessment of the bond strength. In this study, the microtensile bond strength test, preconized by Sano et al,\textsuperscript{26} but without additional wear to obtain an hourglass specimen, was performed in an attempt to eliminate the nonuniform stress distribution within the adhesive zone.\textsuperscript{27} The bonded interfaces of the specimens used in this study are approximately 0.8 mm\textsuperscript{2}, allowing a more uniform stress distribution during loading and failure predominantly at the adhesive interface.

In the present study, three specimens failed cohesively in the ceramic substrate, which is in line with Della Bonna et al\textsuperscript{11} who found that most of the failures in microtensile bond strength tests that evaluate the bond strength between composite resin and ceramic occurred within the adhesion zone.

CONCLUSION

Based on the current results, the following can be concluded:

- Applying a silane-coupling agent in the etched ceramic surface enhanced the microtensile bond strength between the evaluated resin cements and the glass-ceramic surface.
- The self-adhesive resin cement performed as well as the conventional resin cement when bonded to a glass ceramic, regardless of the ceramic surface treatment.

CLINICAL SIGNIFICANCES

The current results indicate that applying one layer of a silane-coupling agent after etching the ceramic surface with hydrofluoric acid 10\% enhanced the bond strength between resin cements and a glass-ceramic surface.

ACKNOWLEDGMENT

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REFERENCES


