The Use of Silane Coupling Agents on Lithium Disilicate Glass Ceramic Repaired with Resin Composite

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Abstract
This study examined the effects of silane coupling agents on lithium disilicate glass ceramic/resin composite shear bond strength. Two silane coupling agents (RelyX ceramic primer, RX and Kerr silane, KS) and adhesive (Single bond2, SB) were used. Lithium disilicate disks were prepared and embedded in dental gypsum. The samples were randomly divided into six groups (n = 10) according to surface treatments: group1 (No tx), group2 (RX), group3 (SB), group4 (KS), group5 (RX+KS) and group6 (RX+SB). Silicone mold was placed on top of treated ceramic surface. Resin composite was filled into mold and then light-cured for 40 seconds. All bonded specimens were kept in distilled water at 37°C for 24 hours and they were subjected to bond strength measurement using a universal testing machine. The data were statistically analyzed using one-way ANOVA and Tukey’s test. The bond strengths of group1 to group6 were 5.13(2.38), 16.36(3.54), 11.14(1.67), 10.49(2.85), 20.84(3.02) and 22.25(2.51) MPa, respectively. Group5 and group6 showed significantly highest bond strength value (P < 0.05). Group2 exhibited significantly higher bond strength values than group3 and group4 (P < 0.05).

In conclusion, the any type of silane had positive effect on lithium disilicate and resin composite interface. Silane application prior to the adhesive material increased bond strength.

Keywords: Adhesive, lithium disilicate glass ceramic, resin composite, silane coupling agent.

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Introduction
An increasing number of lithium disilicate glass ceramic materials are widely accepted and used in dental practice because of its predictable in success rate and excellent esthetic outcome. Lithium disilicate glass ceramic may be broken because of inadequate tooth preparation, trauma, parafunctional habits or occlusal adjustment. Repair of this fracture is conservative treatment. Apart from mechanical retention, e.g. abrasion with aluminum oxide or hydrofluoric acid etch, chemical adhesion using silane coupling agents during repair of lithium disilicate glass ceramic fracture with resin composite.

Silane coupling agents play an important function in the chemical interaction the resin-based material and the lithium disilicate glass ceramics, obtained forming strong siloxane linkages. Trialkoxysialnes, such as 3-methacryloyloxypropyl trimethoxysilane (3-MPS) in most of commonly used in clinical dentistry. Chemical adhesion between the resin-lithium disilicate glass ceramic surfaces can be achieved using a silane coupling agent. Silane is a bifunctional molecule that promotes adhesion via covalent bonds with hydroxyl groups on the lithium disilicate glass ceramic surface. One functional group can react with the inorganic ceramic surface forming stable siloxane networks and the other is a methacrylate group capable of reacting with an organic resin matrix in resin-based materials. The silane application may be one of the most crucial steps in obtaining an optimal bond between silica-based ceramic and resin-based materials.
are commercially available in the dental market. Two-bottle systems are known to provide a longer shelf life and increase initial reactivity compared to the one-bottle system. However, to simplify the bonding procedure, manufacturers tend to produce prehydrolyzed single-bottle silanes that contain other adhesives which may include many other components such as bisphenol A glycidyl methacrylate (Bis-GMA).

The purpose of this study was to investigate the effect of different types of composition of silane coupling agents on the shear bond strength of lithium disilicate glass ceramic and resin composite interface. The null hypothesis of this study was that the type of silane coupling agent used does not affect the shear bond strength of lithium disilicate glass ceramic and resin composite interface.

Materials and methods

The descriptions of the silane coupling agents and adhesive investigated in this study are given in Table 1.

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>RelyX ceramic primer (3M ESPE, St. Paul, Minnesota, USA)</td>
<td>Ethanol, water, methacryloxypropyltrimethoxysilane</td>
</tr>
<tr>
<td>Single bond2 (3M ESPE, St. Paul, Minnesota, USA)</td>
<td>Bis-GMA, HEMA, DMA, methacrylate functional copolymer, filler, photoinitiators, ethanol, water</td>
</tr>
<tr>
<td>Kerr silane (Kerr Corporation, Califonia, USA)</td>
<td>Ethanol, (1-methylethylidene) bis [4,1-phenyleneoxy (2-hydroxy-3,1-propanediyl)] bismethacrylate, Poly (oxy-1,2-ethanediyl), q:q:q:[1-methylene(1,4-phenylene)] bis [4,1-phenyleneoxy (2-hydroxy-3,1-propanediyl)] bis[2,2-ethyleneoxyethyl] dimethacrylate, 3-trimethoxysilylpropyl methacrylate</td>
</tr>
</tbody>
</table>

Table 1. Materials used in this study. Abbreviations: Bis-GMA, bisphenol A-glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; DMA, dimethacrylate.

Preparation of bonding specimens

Sixty lithium disilicate disk specimens (IPS e.max Press, Ivoclar-Vivadent, Schaan Liechtenstein) (10.0 mm in diameter, 5.0 mm in thickness) were supplied by the manufacturer. Each disk specimen was embedded in polyvinyl chloride (PVC) pipe with dental gypsum. The surfaces of all bonding specimens were polished with 600 grid Silicon carbide paper (3M Wetordy abrasive sheet, 3M, Minnesota, USA) followed by ultrasonic cleaning for 10 minutes in distilled water and subsequent drying with triple syringe oil-free air for 10 seconds.

The specimens were randomly divided into 6 groups of 10 specimens each and ceramic surface treated with silane coupling agent and/or adhesive as follows:

- **Group 1**, no ceramic surface treated, serves as a control.
- **Group 2 (RX)**, specimens were treated with RelyX ceramic primer using a microbrush, and then dried with triple syringe oil-free air for 10 seconds.
- **Group 3 (SB)**, specimens were treated with Single bond2 using a microbrush, and then dried with triple syringe oil-free air for 10 seconds, and subsequent light cured 20 seconds.
- **Group 4 (KS)**, specimens were treated with Kerr silane using a microbrush, and then dried with triple syringe oil-free air for 10 seconds, and subsequent light cured 20 seconds.
- **Group 5 (RX+KS)**, specimens were treated with RelyX ceramic primer using a microbrush, and then dried with triple syringe oil-free air for 10 seconds, and subsequent treated with Kerr silane, and then dried with triple syringe oil-free air for 10 seconds, and subsequent light cured 20 seconds.
- **Group 6 (RX+SB)**, specimens were treated with RelyX ceramic primer using a microbrush, and then dried with triple syringe oil-free air for 10 seconds, and subsequent treated with Single bond2, and then dried with triple syringe oil-free air for 10 seconds, and subsequent light cured 20 seconds.

Pieces of polyethylene adhesive tape (Scotch blue painter’s tape, 3M, Minnesota, USA) approximately 80 microns in thickness with 2.0 mm diameter circular holes were placed on the pretreated surfaces of the specimens to define the bonding area. A silicone mold (3.0 mm in diameter, and 2.0 mm in thickness) was placed on the adhesive tape top, and then filled resin composite (Filtex Z350 XT (A3E), 3M ESPE, Dental products, Minnesota, USA), and subsequent light cured 40 seconds (Elipar Freelight 2 LED curing light, intensity 1000 mw/cm², 3M ESPE, Minnesota, USA). The bond specimens were stored at room temperature for 30 minutes, and then were stored in incubator (Contherm 160M, Contherm Scientific Ltd., Korokoro, Lower Hutt, New Zealand) at 37 °C in distilled water for 24 hours.

Shear testing procedure and surface analysis

The shear bond strengths of the specimens were measured in a universal testing machine (AGS-X 500N, Shimadzu corporation,
Kyoto, Japan), with a load applied in the direction parallel to the bonding surface at a crosshead speed of 0.5 mm/minute. The shear bond strength was calculated by dividing the force at which bond failure occurred by the bonding area. The debonded ceramic surfaces were examined under a stereomicroscope (Stereomicroscope; ML 9300, Meiji Techno Co. Ltd., Saitama, Japan) at a magnification of x40 to evaluate failure types. The failure modes were classified as A, an adhesive failure at the ceramic and resin composite interface; B, a mixed failure of an adhesive failure at the ceramic and resin composite interface and a cohesive failure of the resin composite; and C, a cohesive failure in the resin composite.

**Statistical analysis**

The data were statistically analyzed using one-way ANOVA and Tukey’s test with \( P < 0.05 \) was used to indicate significance.

**Results**

The mean bond strength values and standard deviations are shown in Table 2. Group 5 and group 6 showed significantly highest bond strength value \((P < 0.05)\). Group 2 exhibited significantly higher bond strength values than group 3 and group 4 \((P < 0.05)\). Group 1 showed significantly lowest bond strength value \((P < 0.05)\).

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean bond strength (SD)</th>
<th>Failure mode</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Adhesive</td>
<td>Mixed</td>
</tr>
<tr>
<td>No treatment</td>
<td>5.13 (2.36)</td>
<td>100</td>
</tr>
<tr>
<td>RX</td>
<td>16.36 (3.54)</td>
<td>60</td>
</tr>
<tr>
<td>SB</td>
<td>11.14 (1.67)</td>
<td>80</td>
</tr>
<tr>
<td>KS</td>
<td>10.49 (2.85)</td>
<td>90</td>
</tr>
<tr>
<td>RX+KS</td>
<td>20.84 (3.02)</td>
<td>50</td>
</tr>
<tr>
<td>RX+SB</td>
<td>22.25 (2.51)</td>
<td>50</td>
</tr>
</tbody>
</table>

**Table 2. Mean bond strength, standard deviation (Megapascal) and failure mode (%).**

The value with identical letters indicates no statistically significant difference.

The distribution of failure modes after shear bond strength test is summarized in Table 2. All of specimens for group 1 were classified as adhesive failure after fracture. With the silane coupling agents and/or adhesive groups showed mixed failures (10% to 50%), but adhesive failures (50% to 90%). One specimen for group 6 was showed as cohesive failure in resin composite (10%).

**Discussion**

This study examined the effects of silane coupling agents on lithium disilicate glass ceramic/resin composite shear bond strength. The result show significant differences between the shear bond strength of each group. Therefore, the null hypothesis was rejected.

The clinical success of a repaired lithium disilicate glass ceramic depends on the quality and durability of the bond between ceramic and the resin composite. To obtain stable bond strength between lithium disilicate glass ceramic and resin composite, mechanical retention created by surface roughening and chemical adhesion with silane coupling agent is an essential.

In the present study, the negative control group exhibited the lowest shear bond strength as silane coupling agent was not used. All other groups showed significantly higher shear bond strength than the negative control group. However, the shear bond strength varied greatly among the silane coupling agent types. Many factors also may influence the shear bond strength of a material, for example, chemical compositions, temperature variations, or wettability of materials.

RX group showed significantly higher shear bond strength than SB and KS groups. RX group is silane coupling agent. The reason that RX group has higher shear bond strength than SB and KS groups might be due to the pH value at 4.5, creating silanol groups, allowing appropriate chemical interaction with the lithium disilicate glass ceramic and resin composite. KS is silane coupling agent containing primers. Bis-GMA resin monomer, Bis-GMA with silane coupling agent in the same bottle can interfere the condensation reaction of the silane coupling agent with the –OH groups of the lithium disilicate glass ceramic. Moreover, the addition of Bis-GMA resin significantly decreased the efficacy of silane-containing primers. The KS has a pH value of 7.3, the rate of silane hydrolysis is greatly influenced by pH value. Silane can hydrolyze quickly in an alkaline and acidic pH value. However, the process of silane hydrolysis is slowest at neutral pH value for alkoxysilanes. This finding is likely a result of the silane coupling agent presented in KS formulation, allowing improper chemical adhesion with the lithium disilicate glass ceramic and resin.
composite. Thus, the shear bond strength of KS group was inferior compared to RX group.

RX+KS and RX+SB groups showed significantly highest shear bond strength. Silane is also used for repairing lithium disilicate glass ceramics, but only after the prior application of hydrofluoric acid, which produces mechanical retentions on the ceramic surface.\textsuperscript{19} Silane could make the lithium disilicate glass ceramic surface active again and thus able to adhesively interact with the fresh repairing resin composite.\textsuperscript{20} The application of silane prior to the adhesive material increased the shear bond strength of lithium disilicate glass ceramic and resin composite interface.

Regarding the mode of failure, the shear bond strength test can explain predominantly adhesive and mixed failure modes. The adhesive failures were mainly obtained from the control group, RX group, SB group and KS group. The mixed and cohesive failures were often correlated with high shear bond strength, as showed from RX+KS group and RX+SB group (Table 2).

Conclusions

Within the limitations of the present investigation, the any type of silane coupling agent had positive effect on lithium disilicate glass ceramic and resin composite interface. The application of silane coupling agent on lithium disilicate glass ceramic surface prior applied by adhesive material increased the shear bond strength of lithium disilicate glass ceramic repaired with resin composite.

Declaration of Interest

The authors declare no potential conflicts of interest with the materials involved in the present study.

References