

Effect of Different Types of Silane Coupling Agents on the Shear Bond Strength between Lithium Disilicate Glass Ceramic and Resin Cement

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Abstract

This study aimed to evaluate the effect of different types of silane coupling agents on the shear bond strength between lithium disilicate glass ceramic and resin cement. Lithium disilicate disks (IPS e.max Press) were prepared and randomly assigned to six different groups (n=20) based on the type of silane used: control group (no treatment) and five silane-treated groups (Kerr silane primer, Monobond N, RelyX ceramic primer, experimental silane, and Single bond universal). Composite resin rods were luted to the prepared surfaces with resin cement. The samples were stored in distilled water (37°C, 24 hrs) before shear bond strength test was performed. Data were analyzed by one-way ANOVA and Tukey's HSD. RelyX ceramic primer and the experimental silane groups exhibited significantly higher shear bond strengths compared to other groups (p<0.05). The control group showed the lowest bond strength and was significantly different from other groups. It was found that the type of silane coupling agent used significantly influences the bond strength between lithium disilicate and resin cement. Application of any type of silane coupling agent significantly increased the bond strength when compared with the untreated group.

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Introduction

Metal ceramic restorations have been available for more than five decades because of its predictable performance and good esthetic outcome.¹ However, due to a rise in demand to improve esthetics and the trend towards reduction of metal usage and its biocompatibility,² all-ceramic restorations have become more popular.³ The trend for all-ceramic restoration has shifted from layered ceramic to monolithic ceramic to enhance mechanical properties. Monolithic lithium disilicate glass ceramic is one of the most popular silica-based ceramics which provides a good esthetic appearance. Its material also offers superior resistance to chipping and flexural fracture compared with veneered counterparts.⁴

Apart from the properties of the

restorative materials themselves, resin–ceramic bonding greatly influences the clinical outcome.⁵ Reliable resin bonding increases retention, enhances marginal adaptability, minimizes microleakage, and improves fracture resistance. Several methods, including mechanical and chemical modification, have been proposed to achieve the optimal resin–ceramic bond strength. For silica-based ceramic restorations, a reliable bond between the resin and ceramic materials can be achieved by hydrofluoric acid etching (mechanical bonding) along with silane priming (chemical bonding).^{3,6}

Silane coupling agents, which are widely used in the field of dentistry, are known for their excellent performance and biocompatibility. Trialkoxysilanes, such as 3-methacryloyloxypropyl trimethoxysilane (MPS), is one of the commonly used silane coupling agents in dentistry.⁶⁻⁸ Silane molecules can react with water molecules via hydrolysis to produce three silanol groups (-Si-OH) from the corresponding methoxy groups (-Si-O-CH₃). The silanol groups are capable of forming stable siloxane networks (-Si-O-Si-O-) on the glass-ceramic surface.^{6, 8, 9}

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Further, silane also provides an increase in surface energy and wettability of silica-based ceramic.¹⁰ In the case of lithium disilicate ceramic, silane application is one of the most crucial steps in obtaining an optimal bond.¹¹

Many types of silane coupling agents are commercially available in the market. However, to simplify the bonding procedure, manufacturers tend to produce pre-hydrolyzed single-bottle silanes together with universal adhesive primers. These adhesive primers may include many other components such as bisphenol A glycidyl methacrylate (Bis-GMA) and 10-methacryloyloxydecyl dihydrogen phosphate (MDP).¹²

This study investigates the effect of different types of composition of silane coupling agents on the shear bond strength of resin cement to lithium disilicate ceramic. The null hypothesis of this test was that the type of silane coupling agent used does not affect the shear bond strength between lithium disilicate glass ceramic and resin cement.

Materials and methods

Lithium disilicate disks preparation

Table 1 summarizes the materials used in this study. One hundred twenty lithium disilicate disks shade A3 (IPS e.max Press, Ivoclar-Vivadent, Schaan Liechtenstein) were prepared by a wax-up of cylinder-shaped rods (5 mm in diameter and 5 mm in height). Lost wax technique and heat-pressed processing were performed according to the manufacturer's instructions. All specimen disks were embedded into a polyvinyl chloride (PVC) tube with dental gypsum type IV. The ceramic specimens were placed to a depth where their margins were approximately 1 mm higher than the edge of the tube. After the gypsum reached its final setting time, the mounted samples were polished using a polishing machine (Nano 2000 grinder-polisher with FEMTO 1000 polishing head, Pace Technologies, Arizona, USA) with 300 and 600-grit silicon carbide abrasive paper, respectively. The polishing process was performed under running water under a pressure of 2 kg/cm², spinning in a clockwise motion (200 rounds per minute) for 5 minutes. Abrasive papers were replaced between each sample. The samples were ultrasonically cleaned in distilled water for 10 minutes before proceeding to the bonding

procedure. The specimens were etched with 35% phosphoric acid gel (Scotchbond Universal Etchant, 3M ESPE, St. Paul, MN, USA) for 15 seconds, rinsed with distilled water for 15 seconds to remove debris and then air-dried.

Brand name	Type	Composition	Manufacturer/Supplier
IPS e.max Press (Lot Y10318)	Lithium disilicate glass ceramic	SiO ₂ , Li ₂ O, K ₂ O, P ₂ O ₅ , ZrO ₂ , ZnO, other oxides and ceramic pigments	Ivoclar-Vivadent, Schaan, Liechtenstein
Filtek Universal (Lot N929246)	Composite resin	Silane treated ceramic, silane treated silica, diurethane dimethacrylate (UDMA), bisphenol A polyethylene glycol diether dimethacrylate, bisphenol A diglycidyl ether dimethacrylate (BIS-GMA), silane treated zirconia, polyethylene glycol dimethacrylate, triethylene glycol dimethacrylate	3M ESPE, St. Paul, MN, USA
RelyX U200 (Lot 6348451)	Self-adhesive resin cement	Base: Methacrylate monomers containing phosphoric acid groups, Methacrylate monomers, Silanated fillers, Initiator components, Stabilizers, Rheological additives Catalyst: Methacrylate monomers, Alkaline(basic) fillers, Silanated fillers, Initiator components, Stabilizers, Pigments, Rheological additives	3M Deutschland GmbH, Neuss, Germany
RelyX ceramic primer (Lot N988623)	Silane	Ethanol, water, methacryloxypropyltrimethoxysilane	3M ESPE, St. Paul, MN, USA
Monobond N (Lot X41367)	Silane	Ethanol, methacrylated phosphoric acid ester, sulphide methacrylate, 3-trimethoxysilylpropyl methacrylate	Ivoclar-Vivadent, Schaan, Liechtenstein
Kerr Primer (Lot 7072259)	Silane	Ethanol, (1-methylethylidene) Bis [4,1-phenyleneoxy (2-hydroxy-3,1-propanediyl)] bismethacrylate Poly(oxy-1,2-ethanediyl), α,α'-(1-methylethylidene) di-4,1-phenylene] bis [ω-[(2-methyl-1-oxo-2-propen-1-yl)oxy]-2,2'-ethylenedioxydiethyl dimethacrylate 3-trimethoxysilylpropyl methacrylate	Kerr Corp., Orange, CA, USA
Single Bond Universal (Lot 81130A)	Universal adhesive	MDP phosphate monomer, dimethacrylate resins, HEMA, polyalkenoic acid copolymer, fillers, ethanol, water, initiators, silane	3M, Deutschland GmbH, Neuss, Germany
Experimental silane (Lot SHBJ3136)	Silane	Methacryloxypropyltrimethoxysilane, Inhibitors (hindered phenol and/or hydroquinone derivatives), Methanol	Sigma-Aldrich, St. Louis, MO, USA

Table 1. List of materials used.

Composite resin rod preparation

One hundred twenty composite resin rods were prepared using a silicone template (3 mm diameter, 3 mm thick). Composite resin (Filtek Z350 XT (A1), 3M ESPE, St. Paul, MN, USA) was filled and compacted into the silicone mold and then light-irradiated by a light-curing unit (Elipar Freelight 2 LED curing light, 3M ESPE, St. Paul, MN, USA) at 1000 mW/cm² for 40 seconds. The silicone mold was removed from the prepared resin rod and the rod was light-irradiated for another 40 seconds. The silicone mold was wiped clean with ethyl alcohol gauze and air-dried for 30 seconds between each usage.

Setting up the experimental groups

The samples were randomly divided into six groups (n=20) depending on the type of silane coupling agent applied. They are listed as follows:

- Group 1: No silane, serves as a control group
- Group 2: Kerr silane primer

- Group 3: Monobond N
- Group 4: RelyX ceramic primer
- Group 5: Experimental silane
- Group 6: Single bond universal

The experimental silane was prepared with a solution of 95% ethanol and 5% distilled water. The solution was gently mixed in a beaker before calibrating its pH to 4.5 with acetic acid using a digital Orion pH meter (Orion 420A pH meter, Thermo Electron Corp., Beverly, MA, USA). After the pH was calibrated, silane (Sigma-Aldrich, St. Louis, MO, USA) was added to the solution to yield a 2% final concentration. The solution was gently mixed and left untouched for five minutes to allow hydrolysis and silanol formation. The solution was transferred into a plastic bottle with a magnetic bar. The magnetic stirrer (Magnetic stirrer hotplate SM26, Stuart Scientific, Staffordshire, United Kingdom) was used to mix the solution for 1 minute.

Three microliters of the respective silane coupling agent were applied onto the surface of the lithium disilicate specimens via micropipette. Using a micro brush (Citisen Micro Applicator, Huanghua Promisee Dental, Hebei, China), the solution was smeared evenly into a thin coat. Then, the specimens were left untouched, allowing the silane coupling agent to react with the surface of lithium disilicate as recommended by the manufacturer. Any excess coupling agent at or around the border of the specimens was removed using new micro brushes. The specimens were finally air-dried for ten seconds with a pressure of 40-50 pounds per square inch via a triple syringe of a mobile dental unit.

Composite resin rod cementation

To standardize the bonding area of each sample, a one-sided tape was prepared. An 80-micron thick one-sided sticky tape (Scotch blue Painter's tape, 3M ESPE, St. Paul, MN, USA) was cut into a square shape with a size of 10x10 mm. A 2-mm diameter hole was made in the middle of the sticky tape using a hole-puncher. This area represents the bonding site for composite resin. The tape was placed onto the surface of the lithium disilicate glass-ceramic disks after surface treatment. A prepared composite rod was then attached to the bonding site with a thin uniform layer of resin cement (RelyX U200, 3M Deutschland GmbH, Neuss, Germany). A weight of one kilogram was applied

to the composite rod during the bonding procedure using a modified durometer. The excess cement was tack-cured for two seconds on each surface and removed with an explorer. Glycerine gel was applied to the margin of the bonded interface and light-irradiated for 40 seconds to prevent the formation of an oxygen-inhibited layer. The samples were immersed into distilled water at 37°C for 24 hours in an incubator (Contherm 160M, Contherm Scientific Ltd, Korokoro, Lower Hutt, New Zealand) according to ISO/TS 11405 before proceeding to the shear bond strength test.

Shear bond strength test

Each specimen was mounted on the jig of the universal testing machine (EZ-S 500N, Shimadzu Corporation, Kyoto, Japan). The test was conducted with the shearing blade placed on the resin composite at 1.0 mm away and parallel to the bonding site. The load was applied at a crosshead speed of 0.5 mm/min. The shear bond strength (MPa) was calculated by dividing the highest bond strength by the surface area of the resin cement/ceramic interface. The debonded interfaces were viewed under a stereomicroscope (Olympus Stereo Microscopes, SZ61, Tokyo, Japan) at a magnification of ×40 to study the mode of failure. The mode of failure can be classified into four groups:

1. Adhesive failure (AF) that occurs at the interface between the lithium disilicate disk and composite resin. No resin cement is present when the lithium disilicate disk is examined under the stereomicroscope.
2. Cohesive failure within the body of resin cement (CR), where resin cement was seen covering an entire surface of ceramic or there was a fracture of an entire layer of resin cement.
3. Cohesive failure within the body of ceramic (CC), where ceramic was seen covering an entire surface of resin composite or there was a fracture of an entire layer of ceramic.
4. Mixed failure (MF) or a combination of adhesive and cohesive failure, where the ceramic surface demonstrated both characteristics mentioned previously.

Results

According to the statistical analysis result using one-way ANOVA, the bond strength was significantly influenced by the type of silane coupling agent used. Table 2 shows mean shear bond strength values and the distribution of failures. The control group produced the lowest bond strength and was significantly different from other groups ($p < 0.05$). RelyX ceramic primer (RX) and the experimental silane groups exhibited significantly higher shear bond strengths compared with all other groups ($p < 0.05$). The Monobond N group demonstrated significantly higher bond strengths than Kerr silane and Single bond universal groups ($p < 0.05$). However, Kerr silane and Single bond universal groups still presented significantly lower bond strengths than the other groups ($p < 0.05$).

The mode of failure indicates bonding quality. The failed samples were retrieved and observed under a stereoscope at a magnification of 40x. The samples with low bond strengths tended to result in an adhesive failure, while the samples with high bond strengths tended to exhibit mixed failure. All samples from the control group (no silane), Kerr silane, and Single bond universal groups failed adhesively (Table 2). Mixed failures were mostly observed in the Monobond N, RelyX ceramic primer group, and experimental group. No cohesive failure was observed in this study.

Discussion

This study evaluated the effect of different types of silane coupling agents on the shear bond strength between lithium disilicate glass ceramic and resin cement. The results show significant differences between the shear bond strength of each tested group. Therefore, the null hypothesis was rejected.

Surface modification is an important procedure to improve the bond strength of dental materials to a desired substrate.^{13, 14} A clinical recommendation for bonding lithium disilicate ceramic is to etch the bonding surface with 5% hydrofluoric acid for 20 seconds, followed by silane application.^{11, 12, 15-17} Hydrofluoric acid etching induces changes in the glass-ceramic surface morphology, which enhances micromechanical retention. It is proven to be the most successful method in enhancing the bond

strength of lithium disilicate glass-ceramic.^{11, 17-20} However, in this study, hydrofluoric acid was not used prior to the application of silane coupling agents since the aim was to focus on the effect of silane coupling agents on the chemical bond strength of lithium disilicate and resin cement alone. Previous studies^{12, 16, 21-24} demonstrated that the application of silane alone without hydrofluoric acid etching can significantly increase the bond strength of lithium disilicate ceramic.

Surface treatments	Mean \pm SD (MPa)	Mode of failure (AF/CR/CC/MF)
No silane	4.16 \pm 0.95 ^A	20/0/0/0
Kerr silane primer	7.23 \pm 1.15 ^B	20/0/0/0
Monobond N	15.26 \pm 2.24 ^C	6/0/0/14
RelyX ceramic primer	20.97 \pm 2.33 ^D	2/0/0/18
Experimental silane	22.76 \pm 2.40 ^D	1/0/0/19
Single bond universal	6.95 \pm 0.94 ^B	20/0/0/0

Table 2. The means and standard deviations of shear bond strength and mode of failure. The same superscript indicates no significant difference. Abbreviations: SD, standard deviation.

In this study, the control group demonstrated the lowest bonding ability as silane was not applied. Consequently, no strong siloxane linkages were formed between silane and the substrate.⁶ All other groups demonstrated significantly higher shear bond strength than the control group. However, the bonding ability varied greatly among the type of silane used. This variation might be due to multiple factors that may influence the bond strength of a material, such as variations in chemical composition, wetting ability, viscosity, or the properties of the resin cement.²²

Furthermore, other factors may influence the rate of hydrolysis of silane, which may also contribute to the outcome of the bond strength, such as silane molecular structure, the concentration of silane, pH, temperature, humidity, and solvent system.⁶ When silane is applied onto the surface of lithium disilicate ceramic, the surface becomes more hydrophobic, which is beneficial as it increases the wettability of composite/resin material.^{25, 26}

RelyX ceramic primer group and the experimental silane group demonstrated significantly higher bond strength than other groups. One reason that both silane groups yielded higher bond strength compared with

other groups might be due to the pH value of the solution. The hydrolysis reaction of silane can best occur at a pH value of about 4.5, forming silanol groups.^{6,8}

The Monobond N group offered significantly higher bond strength than Kerr silane, Single bond universal group, and control group; however, its bond strength was lesser than that of the RelyX ceramic and Experimental groups. Our results are similar to Taira et al.'s²⁷ study; in their study, various silane primers were used to enhance the bond strength between a ceramic block and resin composite bonded with dual-curing luting agent. The mean shear bond strength of the Monobond plus group was significantly lower than the conventional silane primers but significantly higher than the unprimed specimens. Monobond N has a pH value of 1.63.²⁸ According to the manufacturer, the primer is composed of silane methacrylate, phosphorus-containing monomer, and other sulfur-containing monomers, which together make it capable of bonding to glass-ceramic, oxide ceramic, metal, composite, and fiber-reinforced composites. Nevertheless, the phosphorus monomer may cause a change in pH, which might affect the rate of hydrolysis of silane monomer and may impair the stability of functional silane.²⁹

In this study, Kerr silane primer, silane containing resin-monomer and Single bond universal adhesive, offered the least strength compared with other groups of silane coupling agent but was still significantly higher than the negative control, even though the wettability of the resin was improved.³⁰ Kerr silane has a pH value of 7.3. The resin monomers are added presumably to eliminate the further application of unfilled resin to the silanated surface prior to the application of resin cement. According to Chen et al.,³⁰ incorporating Bis-GMA resin into a silane solution significantly reduces the contact angle and bond strength. The additional resin may inhibit the chemical reaction between the silane primer and lithium disilicate ceramic. Kerr silane demonstrated similar contact angle to that of unprimed lithium disilicate surface, which means the chemical bonding that the silane offered is limited or nonexistent. The low contact angle suggested that the extra resin may interfere with the condensation process of forming the siloxane bond by inhibiting the liberation of water molecules. Moreover, the rate of silane hydrolysis is strongly influenced by pH. Silane

can hydrolyze faster in an acidic and alkaline environment. However, the hydrolysis process is slowest at neutral pH for alkoxy-silanes.⁶

The application of a silane coupling agent as a separate step tends to be more effective in producing optimal ceramic-resin cement bonding than those that are contained in universal adhesive. Many researchers have confirmed that the silane coupling agent contained into universal adhesives do not appear to enhance the bond strength between glass ceramic and resin cement.^{16, 31, 32} This might be because the stability of silane coupling agents in universal adhesives is lower in an acidic environment (pH 2.2-3.2).³² The presence of different types of monomers in universal adhesive may affect the bonding capability of silane coupling agent within the adhesive, for example, the presence of Bis-GMA may interfere with the condensation reaction between silanol group and the substrate.^{31, 32} In addition, the concentration of silane in universal adhesive may not be sufficient to chemically react with the hydroxyl group on the ceramic surface which may produce inadequate bond quality.^{31, 32}

The mode of failure that occurred in this study were adhesive and mixed failures. The adhesive failures were mainly correlated with low shear bond strength, as obtained from the control group, Kerr silane group, and Single bond universal group. It is considered that the bonding force at the ceramic/cement interface is lower than the cohesive strength on ceramic and resin cement. On the contrary, mixed failures were often correlated with high shear bond strength value, as obtained from the other groups.

There are possible limitations of this study: since it is an in vitro study, only one type of resin cement was used. Another possible limitation is that the tests were performed immediately after the application of the adhesive. RelyX U200, a self-adhesive resin cement, was the only cement used in this study. Since it was an in vitro model, the model can only offer monotonic loading which is different from cyclic fatigue forces that are generated intraorally. Finally, when the tests were performed post-application immediately, the longevity of the bond was not tested.

Further studies may require using different types of silane coupling agents with different compositions, and long-term bonding stability should be performed (e.g.,

thermocycling). It can be concluded that treating the ceramic surfaces with silane coupling agents that contain other components other than MPS alone may yield lower bond strength value than ones without additives. Therefore, silane coupling agents that contain additives may be easy to use but might also reduce the bond strength. This information may be helpful for the dentists to aid in selecting a proper silane coupling agent suitable for each case to provide long-term success.

Conclusions

In conclusion, the type of silane coupling agent used significantly influences the bond strength between lithium disilicate and resin cement. The application of any type of silane coupling agent significantly increases the bond strength when compared with the untreated group; however, the bond strengths vary in accordance with the type of silane used. Differences in bond strength depend on several other factors.

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Declaration of Interest

The authors declare no conflict of interest.

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