Bond strength of resin cement to ceramic with simplified primers and pretreatment solutions

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Manufacturers have recently introduced surface primers and pretreatment solutions that reportedly simplify the bonding process of resin cements to ceramics through various combinations of etchant and coupling agents. This study evaluated the shear bond strength (SBS) of a resin cement to a lithium disilicate glass-ceramic material pretreated with various new surface treatment solutions and compared the results to those of a control group prepared with the traditional application of hydrofluoric acid (HF) and silane. Resin cement was bonded to pretreated glass-ceramic surfaces, and specimens were tested for SBS after 24 hours of storage in water. Traditional surface treatment of lithium disilicate glass ceramic with HF and silane resulted in a significantly greater mean SBS than did simplified primer solutions. There were no statistically significant differences among the simplified pretreatment groups. In the control group, the majority of failures were due to mixed adhesive-cohesive fracture, while in the simplified treatment groups the failure mode was usually adhesive, suggesting a weaker interface.

Received: May 17, 2016
Accepted: July 6, 2016

GENERAL DENTISTRY SELF-INSTRUCTION CE CREDIT
Exercise No. 427, p. 38
Subject code: Basic Science (010)

All-ceramic restorations have become a favorable alternative to metal-ceramic and all-metal restorations as more and more patients request enhanced esthetic results.[1] Ceramics can be divided into different categories based on the materials used in their fabrication.[2] McLaren & Cao divided dental ceramics into 4 composition categories.[2] Category 1 materials are glass-based systems that contain mainly silica dioxide (either silica or quartz) and varying amounts of alumina. These systems are typically powder-liquid versions that can be used to create porcelain veneers and fine-grained machinable blocks for use in computer-aided design/computer-aided manufacturing systems.[2,3]

Category 2 materials are also glass-based systems, usually with silica plus crystalline fillers. These fillers include fluorapatite, leucite, and/or lithium disilicate. The addition of different fillers or different amounts of individual fillers changes the properties of the ceramic.[2] Leucite-reinforced materials may be milled, pressed, or used in a powder-liquid system. Lithium disilicate materials contain a similar glass base as those with leucite fillers but with the use of lithium disilicate crystals.[2] However, because of the unique shape of lithium disilicate crystals, the flexural strength and fracture toughness are significantly increased compared to leucite-reinforced materials, while the lithium silicate fillers still retain a high level of translucency, making lithium disilicate materials suitable for both anterior and posterior applications. The lithium disilicate materials also come in both pressable and millable forms.[3]

Category 3 includes ceramic materials that are crystalline-based systems with glass fillers. The crystalline structure (eg, alumina, alumina-zirconia, or alumina-magnesium) is then infiltrated with lanthanum glass, and the materials can be used to fabricate restorations either by a process known as *slip casting* or by milling.[2,3] These infiltrated ceramics have a high degree of flexural strength, which is attributed to the high amount of crystals in the structure.

Category 4 materials consist of polycrystalline solids of either aluminum or zirconium oxides without a glass matrix. These oxide ceramics have high strength and fracture toughness but less translucency than other fillers.[1-4]

In categories 1 and 2, the glass-ceramic materials may be chemically etched, silanated, and bonded with resin-based cements to maximize adhesion and strength. Etching with hydrofluoric acid (HF) provides an irregular, retentive surface, while silane provides the chemical bond between the matrix of the composite resin and the silica in the glass. However, HF is considered a hazardous substance; thus, a safer, simplified etching or conditioning process could be considered to be clinically advantageous.[5]

Because of their intrinsic strength, the oxide ceramic materials in categories 3 and 4 do not require adhesive cementation.[6]

However, in cases of compromised retention—such as teeth with short clinical crowns—a more durable bond to an oxide ceramic may be preferable. The densely sintered oxide ceramics have surface structures with little to no glass phase and therefore require alternative techniques for bonding.[7] Several methods of surface treatments and modifications have been proposed to increase the retention of oxide ceramics.[7-11] Roughening the intaglio surface with air abrasion can increase surface roughness but may introduce microcracks and cause phase transformation, resulting in a reduction in the strength of the ceramic.[8] However, there are no controlled clinical studies evaluating the effects of airborne particle abrasion on oxide ceramics.[7] Manufacturers have marketed different cement systems and primers that contain functional monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), that provide a chemical adhesion to alumina and zirconia. Several other techniques also have been utilized, such as tribochemical coating, plasma spraying, and selective infiltration etching.[9-11] However, the literature suggests that establishing a strong and reliable bond to oxide ceramics (especially zirconium oxide) is often difficult and unpredictable.[12]

New surface treatment products that claim to make the cementation process faster, easier, and safer than previous methods have been introduced to the market. The manufacturers of these products have attempted to combine separate steps (such as etchant and silane) or combine primers specific to the different types of ceramics (such as 10-MDP combined with silane). However, laboratory studies have suggested that the mix of acidic primers with silane may render the silane less effective in the bonding process.[13,14]

According to the manufacturer, Interface (Apex Dental Materials) is a "…revolutionary ceramic primer, which allows the clinician to bond any type of ceramic to a tooth, including new higher strength materials."[15] The material is a proprietary blend of organic and inorganic acids and silane.[15] The manufacturer further claims that this product can replace HF etchant and silane for bonding with glass-based ceramics or serve as the primer with oxide-based ceramics.[15]

Monobond Plus (Ivoclar Vivadent) is a simplified system, marketed as a universal restorative primer, that combines 3 different functional groups: silane, 10-MDP, and disulfide acrylate.[16] This universal primer reportedly aids in the adhesive bond between luting cement and all indirect restorative materials, including glass or oxide ceramics and metals.[16]

The manufacturer recently released Monobond Etch & Prime (Ivoclar Vivadent) for use with glass ceramics.[17] This product reportedly "etches and silanizes silicate surfaces in 1 easy working step" without the use of HF.[17] The manufacturer suggests that this product creates a durable bond with glass ceramics, comparable to that achieved with an HF etchant and silane process.[17]

OptiBond XTR (Kerr Corporation) is a self-etching universal adhesive for use with direct and indirect restorations.[18] The manufacturer suggests that the use of silane is optional in the bonding of glass ceramics when NX3 resin cement (Kerr Corporation) is used. The instructions for use include air abrasion with aluminum oxide and etching with HF.[18]

To date, little research has been published evaluating these new simplified combination surface primers and solutions. The purpose of the present study was to compare the shear bond strength (SBS) of resin cement to a lithium disilicate glass-ceramic material after the use of new surface primers and pretreatment solutions. The null hypothesis was that there would be no difference in the SBS of resin cement to lithium disilicate based on the type of surface pretreatment solution or primer.

Materials and methods

Lithium disilicate blocks (e.max CAD, Ivoclar Vivadent) were sectioned into 3-mm-thick block specimens with a precision saw (IsoMet 5000, Buehler) and then crystallized in a ceramic oven (Programat P500, Ivoclar Vivadent) according to the manufacturer's instructions. Next, the ceramic specimens were mounted in 1-inch polyvinyl chloride pipes with dental stone. The surfaces of the specimens were steam cleaned and air dried.

Eighty specimens were divided into 5 groups (n = 16) based on the ceramic surface preparation: 1, Interface; 2, Monobond Plus; 3, Monobond Etch & Prime; 4, OptiBond XTR; and 5, Bis-Silane (Bisco). Table 1 describes the compositions and application procedures of the tested materials.

Surface treatment

Group 1

Lithium disilicate specimens were air abraded with 50-μm aluminum oxide (Quattro IS, Renfert USA) according to the manufacturer's recommendations for Interface. The distance of the air-abrasion tip from the ceramic surface was kept at 10 mm with the use of a simple positioning support jig. Interface was then mixed and applied.

Group 2

A 4.8% HF gel (IPS Ceramic Etching Gel, Ivoclar Vivadent) was applied to the lithium disilicate specimens, which were then rinsed and air dried. Monobond Plus was applied in accordance with the manufacturer's instructions.

Group 3

Monobond Etch & Prime was applied to the surface of the lithium disilicate specimens as described in the manufacturer's instructions.

Group 4

Lithium disilicate specimens were air abraded with 50-μm aluminum oxide according to the manufacturer's recommendations for OptiBond XTR. Then HF was applied as in group 2, and specimens were rinsed and air dried. OptiBond XTR was applied according to the manufacturer's instructions.

Group 5 (control)

An HF gel was applied as in group 2, and then specimens were rinsed and air dried. Bis-Silane was applied in accordance with the manufacturer's instructions.

Resin cement bonding

The specimens were then mounted in a jig (Ultradent Products). Automixed dual-cure NX3 resin cement was injected into a white, nonstick polyacetal mold to a height of 4 mm and light cured for 20 seconds with a Bluephase G2 (Ivoclar Vivadent)

Table 1. Surface treatments used in the present study to pretreat lithium disilicate.

Group	Product	Composition	Application
1	Interface	Proprietary blend of organic and inorganic acids and (trimethoxysilyl)propyl methacrylate (silane)	The lithium disilicate specimens were air abraded with 50-µm aluminum oxide (per the manufacturer's instructions) at 30 psi for 10 s, steam cleaned, and air dried. Interface was prepared by mixing 1 drop from bottles A and B. The 2 liquids formed a bubble, which collapsed after 20-30 s. The mixture was then stirred for 5 s with a microbrush applicator, and an even coat was applied to the specimens. The specimens were left to set for 10 s and then air dried for 5 s.
2	Monobond Plus	Ethanol, silane, 10-MDP, and disulfide acrylate	HF was applied for 20 s, and then the specimens were rinsed and air dried for 30 s. One coat of Monobond Plus was applied and left for 60 s, and then the specimens were air dried for 5 s.
3	Monobond Etch & Prime	Silane, ammonium polyfluoride (etchant), alcohol, and water	Monobond Etch & Prime was applied with microbrush, agitated for 20 s, and allowed to sit for another 40 s. The specimens were rinsed with water and air dried for 10 s.
4	OptiBond XTR	*Primer:* glycerophosphate dimethacrylate, hydrophilic comonomers, water, ethanol, and acetone *Bond:* resin monomers, hydroxyethyl methacrylate, inorganic fillers, ethanol, and photoinitiators	The lithium disilicate specimens were air abraded with 50-µm aluminum oxide at 30 psi for 10 s, steam cleaned, and air dried. HF was applied for 20 s, and the specimens were rinsed and air dried for 30 s. One coat of OptiBond XTR was applied and air dried, first gently and then with greater force, to prevent pooling. The specimens were then light cured for 10 s.
5	Bis-Silane	Ethanol and silane	Bis-Silane was prepared by mixing 1 drop each from bottles A and B with a microbrush. Two coats were brushed on the HF-etched lithium disilicate surface. After 30 s, specimens were dried with an air syringe.

Abbreviations: 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; HF, hydrofluoric acid; NA, not applicable.

light-curing unit. The irradiance of the curing light was determined with a radiometer (LED Radiometer, Kerr Corporation) to verify that the levels were above 1000 mW/cm^2.

The specimens were stored in 37°C distilled water in a laboratory oven (model 20GC, Quincy Labs) for 24 hours.

Fracture testing

After 24 hours of storage, the specimens were loaded perpendicular to a knife-edged blade in a universal testing machine (model 5943, Instron). A shear force was applied at a crosshead speed of 1 mm/min until failure.

The SBS values (in megapascals) were calculated by dividing the peak load of failure by the specimen surface area. A mean and standard deviation were determined for each group. Due to the nonnormal distribution of some of the groups, as well as unequal variances, the data were analyzed with nonparametric tests. Kruskal-Wallis and Mann-Whitney U (with a Bonferroni correction) tests were used to evaluate the effect of the surface pretreatment on the SBS of the resin cement to ceramic ($\alpha = 0.005$).

Following testing, each specimen was examined using a 10× stereomicroscope to determine if the failure mode was an adhesive fracture at the resin cement–ceramic interface, a cohesive fracture in the resin cement, a mixed (combined adhesive and cohesive) fracture in the resin cement or ceramic, or a cohesive fracture in the ceramic.

Results

A significant difference in the SBS values of resin cement to lithium disilicate based on the type of surface treatment was found ($P < 0.001$). The control group pretreated with HF and silane had the highest mean (SD) SBS, 23.2 (7.2) MPa, which was significantly greater than the SBS of all the other groups. Monobond Etch & Prime had the lowest mean (SD) SBS, 8.0 (7.4) MPa, but the value was not significantly different from the means of the other simplified primer solutions (Table 2). Fewer adhesive failures were observed in the HF and silane control group (Chart).

Discussion

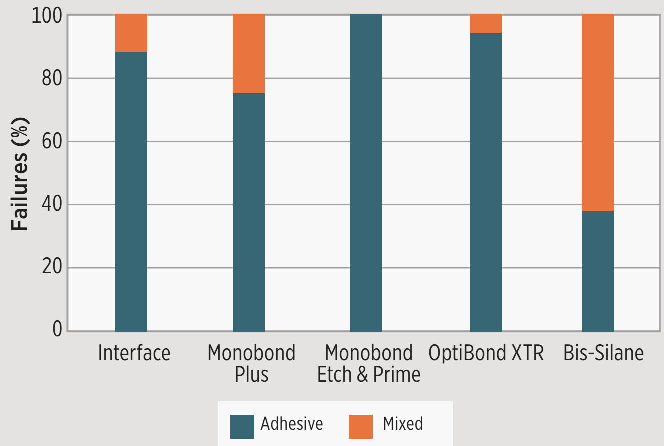
The null hypothesis was rejected, as a significant difference in the SBS values of the resin cement to lithium disilicate was found, and the difference was based on the specific surface pretreatment primer or solution. In this study, all the novel surface pretreatments performed similarly to one another, resulting in no statistically significant differences in mean SBS values. However, the combined or simplified solutions resulted

Table 2. Shear bond strengths (MPa) of resin cement to pretreated lithium disilicate (n = 16 per group).

Group	Surface pretreatment	Mean (SD)
1	Interface	8.4 (3.9)[b]
2	Monobond Plus	11.7 (6.0)[b]
3	Monobond Etch & Prime	8.0 (7.4)[b]
4	OptiBond XTR	10.6 (4.4)[b]
5	Bis-Silane (control)	23.2 (7.2)[a]

Groups with the same lowercase superscript letter are not significantly different ($P > 0.07$).

Chart. Failure modes between resin cement and pretreated lithium disilicate (n = 16 per group).



in significantly lower SBS values of resin cement to lithium disilicate when compared to the control group, which was pretreated with HF etchant and silane. When the modes of failure were evaluated, the use of HF and silane resulted in more mixed adhesive-cohesive failures. However, all of the novel surface pretreatments resulted in predominantly adhesive failures to the lithium disilicate, suggesting a weaker interface.[19]

Interface is a 2-bottle primer system marketed for the preparation of any type of ceramic as well as enamel and dentin. The composition of Interface is largely proprietary; however, bottle A contains a blend of organic and inorganic acids, while bottle B contains (trimethoxysilyl)propyl methacrylate (silane).[15] The 2 solutions remain separate until used. Interface contains novel acids with silane primers for preparing glass-ceramic surfaces without the use of a separate (potentially hazardous) HF etchant.[15] However, the mean SBS value of Interface was less than that of the control group in the present study. It is possible that the organic and inorganic acids did not sufficiently roughen the glass-ceramic surface.

Monobond Plus is considered a universal primer system containing 3 different functional methacrylates—silane methacrylate (glass ceramics), phosphoric methacrylate (oxide ceramics), and sulfide methacrylate (metal)—for bonding to all dental material substrates.[16] The use of HF is recommended when this product is being used to bond resin cement to glass ceramics. In this study, the SBS values of Monobond Plus were less than the control group of HF etchant and silane. The acidic nature of phosphate monomers—such as the 10-MDP used in Monobond Plus—may have reduced the function of the silane.[14] The silane may also have been hydrolyzed, resulting in the reduction of its priming ability due to a condensation reaction, resulting in a polysiloxane oligomer.[20]

In a recent study by Lise et al, Monobond Plus produced higher SBS values to lithium disilicate compared to no treatment or treatment with HF only.[21] However, Monobond Plus was not compared to applications using HF and silane-only solutions, as was done in the present study. A study by Yi et al evaluated the SBS values of Monobond Plus for bonding to zirconia.[13] The authors concluded that use of the primer Z-Prime Plus (Bisco) with air abrasion resulted in higher SBS values than Monobond Plus with air abrasion.[13] Similarly, Kobes & Vandewalle found that SBS values of resin cement (MultiLink Automix, Ivoclar Vivadent) to zirconia were significantly higher with Z-Prime Plus than with Monobond Plus, Clearfil Ceramic Primer (Kuraray America), and AZ Primer (Shofu Dental).[22] Z-Prime Plus contains only 10-MDP and carboxylate monomers, while Monobond Plus and Clearfil Ceramic Primer contain both 10-MDP and silane.[14] The manufacturer did not include silane in Z-Prime Plus; the greater concentration of 10-MDP may facilitate greater SBS values between the resin cement and zirconia.

Monobond Etch & Prime has been marketed as a 1-step, self-etching surface pretreatment for glass ceramics that contains a new polyfluoride conditioner and silane in a single bottle. The manufacturer sought to eliminate the use of an HF etchant and simplify the process to a 1-step procedure, similar to that of Interface.[17] According to the manufacturer, the polyfluoride creates a roughness pattern on the ceramic that is less pronounced than that created by HF but just as efficient for bonding. In the present study, the mean SBS value of resin cement to lithium disilicate was significantly less when Monobond Etch & Prime was used than when HF and silane were used (control group). The polyfluoride conditioner may not have sufficiently roughened the glass-ceramic surface or it may have reduced the efficacy of the silane.

OptiBond XTR is a 2-step, self-etch, light-cured, universal dental adhesive. The self-etching primer contains an acidic phosphate monomer, glycerophosphate dimethacrylate, which reportedly provides chemical and mechanical adhesion with any ceramic material or any composite resin or core material.[18] According to the manufacturer, when OptiBond XTR is used for bonding to glass ceramics, the use of silane is optional (after air abrasion with aluminum oxide and etching with HF) if NX3 resin cement is used. The present study used NX3, which is a dual-cure esthetic resin cement that does not contain additional functional monomers. The SBS values of the resin cement to lithium disilicate were significantly less with OptiBond XTR compared to the control group of HF and silane.

A recent study evaluated the SBS values of resin cement to lithium disilicate when Scotchbond Universal (3M ESPE) adhesive was used with or without the prior use of silane.[23] Scotchbond Universal contains silane, and the manufacturer suggests that additional silane is not required for bonding to glass ceramics. Nevertheless, lower SBS values were found when the universal adhesive was placed without the additional use of silane.[23] According to the authors, the constituent silane in the universal adhesive was not effective in optimizing the ceramic-resin bond. A recent study by Passia et al determined that universal bonding agents that do not contain silane (eg, OptiBond XTR) provide significantly reduced SBS values of resin cement to lithium disilicate compared to a silane-containing primer solution (eg, Monobond Plus).[24]

A large base of published evidence supports the use of traditional surface treatment with HF and silane for bonding to glass ceramics.[2,7,25-27] Tian et al conducted a literature review elucidating the role of HF and silane in the bonding procedure to lithium disilicate glass ceramic.[25] The bifunctional monomer creates a durable bond to silica in both the ceramic and the resin cement.[2,7,26] The silane coupling agent used in the present study (Bis-Silane) does not contain any additional functional monomers. Bis-Silane is a 2-part silane coupling agent that reportedly offers additional shelf-life stability.[27]

Although new surface treatment products that attempt to combine separate steps (such as HF etchant and silane) or combine primers specific to the different types of ceramics (such as 10-MDP combined with silane) have been introduced, few evaluations of these products have been published. Studies that have evaluated these combination products have rarely compared them to a control group pretreated with HF etchant and silane alone (ie, silane not mixed with other primers).[21,24] Given the results of the present study, clinical research is indicated to determine whether these simplified or combination primers and pretreatments should be routinely used by practitioners.

Conclusion

Traditional surface treatment of lithium disilicate glass ceramic with HF etchant and silane resulted in a significantly greater mean SBS to resin cement than did treatment with simplified primers and pretreatment solutions.

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Acknowledgment

Funding for this study was provided by the 59th Clinical Research Training Division, Joint Base San Antonio - Lackland, Texas.

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The views expressed in this article are those of the authors and do not reflect the official policy of the US Air Force, the Department of Defense, Uniformed Services University of the Health Sciences, or the US government. The authors report no conflicts of interest pertaining to any of the products or companies discussed in this article.

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