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Influence of Etching Protocol and Silane Treatment with a Universal Adhesive on Lithium Disilicate Bond Strength

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Clinical Relevance

Optimal bond strength to lithium disilicate is achieved by exposure to at least 20 seconds of 5% hydrofluoric acid (HF) followed by a coat of silane and then a universal adhesive. If an additional silane step is not taken prior to applying a universal adhesive, the use of 9.5% HF for 60 seconds can increase bond strength.

SUMMARY

Objectives: To measure the effects of hydrofluoric acid (HF) etching and silane prior to the application of a universal adhesive on the bond strength between lithium disilicate and a resin.

Methods and Materials: Sixty blocks of lithium disilicate (e.max CAD, Ivoclar Vivadent) were sectioned into coupons and polished. Specimens were divided into six groups (n=10) based on surface pretreatments, as follows: 1) no treatment (control); 2) 5% HF etch for 20 seconds (5HF); 3) 9.5% HF etch for 60 seconds (9.5HF); 4) silane with no HF (S); 5) 5% HF for 20 seconds + silane (5HFS); and 6) 9.5% HF for 60 seconds + silane (9.5HFS). All etching was followed by rinsing, and all silane was applied in one coat for 20 seconds and then dried. The universal adhesive (Scotchbond Universal, 3M ESPE) was applied onto the pretreated ceramic surface, air thinned, and light cured for 10 seconds. A 1.5-mm-diameter plastic tube filled with Z100 composite (3M ESPE) was applied over the bonded ceramic surface and light cured for 20 seconds on all four sides. The specimens were thermocycled for 10,000 cycles (5°C-50°C/15 s dwell time). Specimens were loaded until failure using a universal testing machine at a crosshead speed of 1 mm/min. The peak failure load was used to calculate the shear bond strength. Scanning electron microscopy images were taken of representative e.max specimens from each group.

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Results: A two-way analysis of variance (ANOVA) determined that there were significant differences between HF etching, silane treatment, and the interaction between HF and silane treatment ($p<0.01$). Silane treatment provided higher shear bond strength regardless of the use or concentration of the HF etchant. Individual one-way ANOVA and Tukey post hoc analyses were performed for each silane group. Shear bond strength values for each etch time were significantly different ($p<0.01$) and could be divided into significantly different groups based on silane treatment: no silane treatment: 0 HF < 5% HF < 9.5% HF; and RelyX silane treatment: 0 HF < 5% HF and 9.5% HF.

Conclusions: Both HF and silane treatment significantly improved the bond strength between resin and lithium disilicate when used with a universal adhesive.

INTRODUCTION

Lithium disilicate is a dental ceramic that mimics the esthetics and strength of natural tooth structure. The 70% crystal phase of this unique glass-ceramic material refracts light naturally and provides superior structural reinforcement, imparting a greater flexural strength than is associated with traditional feldspathic porcelain or leucite-reinforced glass ceramics.[1,2] Lithium disilicate crowns may be placed by either traditional cementation techniques or adhesive bonding. One clinical trial has shown similar survival of lithium disilicate crowns cemented with resin-modified glass ionomer cements and bonded with resin cements.[3,4] In clinical situations involving short clinical crowns or overtapering of the crown preparation, adhesive bonding is recommended.[5] *In vitro* studies[6,7] have shown superior bond strength when lithium disilicate is bonded to tooth as compared to traditional cementation. Additionally, bonding with a resin composite cement may also improve the fracture strength of lithium disilicate crowns.[8]

Prior to bonding lithium disilicate crowns, etching with hydrofluoric acid (HF) is recommended.[9] The action of HF etching on the microstructure of these ceramics is by dissolution of the glassy phases of porcelain.[5,6] This phase is partially dissolved to create an appropriate microstructure that increases surface area for bonding.[7-10] Studies[11-15] have shown that HF etching with lithium disilicate improved bond strength. These studies have etched with 9.5%-10% HF for 60 seconds[13,15] or 20 seconds[11,12] and with 5% HF for 20 seconds.[14] Since there has not been an evaluation on the effect of HF concentration on bond strength, this study will compare two reported etching protocols: 9.5% HF for 60 seconds[13,15] and 5% HF for 20 seconds.[14] Silanes are a class of organic molecules that contain one or more silicon atoms. The specific silane used in dentistry is 3-methacryloxypropyltrimethoxysilane. It is used as a chemical coupler, linking organics (resin-based materials) to inorganics (eg, porcelain, some oxidized metals, and glass fillers in resin-based composites).[16] Infrared spectroscopy has shown that silane has the potential to react with hydroxyl (-OH) groups present on the surface of silica in ceramics and the methacrylate group of a bonding agent or resin cement.[17,18] A study by Panah and others[15] demonstrated that the bond strength to lithium disilicate was significantly improved with silane treatment and further improved with HF etching and then use of silane. Nagai and others[14] concluded that silane improved the bond strength to lithium disilicate. Additionally, they discovered that HF etching improved bond strength more in unsilanated specimens than in those that were silanated.[14]

The introduction of universal adhesives presents a new simplified approach for bonding ceramic to resin cements. Universal adhesives contain silane and a monomer called 10-methacryloxydecyl dihydrogen phosphate (MDP) that help bond ceramic to the resin in a cement. A study by Amaral and others[19] has shown the ability of a universal adhesive to bond zirconia to a resin; however, the effectiveness of the adhesive has not been thoroughly investigated with lithium disilicate.

This study examined how surface treatment (HF and silane) affects shear bond strength between a resin composite and lithium disilicate treated with a universal adhesive (Scotchbond Universal, 3M ESPE, St Paul, MN, USA). The purpose of this study was to determine if it is necessary to apply silane prior to a universal adhesive. Additionally, we wished to determine if HF etching improves bond strength to lithium disilicate and to compare the effectiveness of 5% HF (20 seconds) and 9.5% HF (60 seconds). The null hypotheses of this study were that there is no difference in the bond strength when silane is applied prior to a universal adhesive and that there is no difference in bond strength with two different etching protocols prior to a universal adhesive application.

Table 1: *Materials Used in this Study*

Material	Brand Name	Manufacturer	Lot No.
Lithium disilicate	e.max	Ivoclar Vivadent	—
5% Hydrofluoric acid etchant	Ceramic etching gel	Ivoclar Vivadent	R05638
9.5% Hydrofluoric acid etchant	Porcelain etchant	Bisco	1200006564
Silane	RelyX Ceramic Primer	3M ESPE	N371615
Universal adhesive	Scotchbond Universal	3M ESPE	472585
Resin composite	Z100	3M ESPE	N352896

METHODS AND MATERIALS

All materials used for specimen preparation are described in Table 1. Blocks of lithium disilicate (e.max CAD, Ivoclar Vivadent, Amherst, NY, USA) in bisque (blue, metasilicate) form were sectioned into rectangular coupons using a low-speed cutting device (Isomet, Buehler Ltd, Lake Bluff, IL, USA) and sintered according to the recommended protocol. To establish a uniform surface, each specimen was polished with a rotational polishing device using 180- and 320-grit silica carbide abrasive paper under a steady stream of water. The polishing was done by rotating the specimen 90° every one minute with each grit, for a total of four minutes. The specimens were finished with 0.5-μm Al_2O_3 slurry, rotating the specimens 90° every 30 seconds for a total of two minutes. All specimens were subjected to ultrasonic cleaning in distilled water for 15 seconds.

The specimens were divided into groups (n=10) based on surface pretreatments, as follows (Table 2): 1) no treatment (control); 2) 5% HF etch for 20 seconds (5HF); 3) 9.5% HF etch for 60 seconds (9.5HF); 4) silane (S); 5) 5% HF for 20 seconds + silane (5HFS); and 6) 9.5% HF for 60 seconds + silane (9.5HFS).

Groups 5HF, 9.5HF, 5HFS, and 9.5HFS were etched with HF. For the etching procedure using 5% HF (Ceramic etching gel, Ivoclar), a drop of etchant was evenly spread for 20 seconds over the bonding surface of the ceramic using a microbrush. The surface was cleaned with water for 10 seconds. The 9.5% HF (Porcelain etchant, Bisco, Schaumburg, IL, USA) was applied for 60 seconds and cleaned with water for 10 seconds. Groups S, 5HFS, and 9.5HFS then received one coat of silane (RelyX Ceramic Primer, 3M ESPE), which was applied for 20 seconds using a microbrush and then air dried for 10 seconds with room-temperature air. Scotchbond Universal adhesive (3M ESPE) was applied onto all pretreated ceramic surfaces for 20 seconds using a microbrush, followed by air thinning for 10 seconds. The adhesive was cured for 10 seconds with an LED curing light (1200 mW/cm^2; Elipar S10, 3M ESPE). Curing light output was tested after finishing every 10 specimens to check for uniformity of light output using a radiometer (Power Max, Molectron Detector Inc, Portland, OR, USA).

A 5-mm-long, transparent, plastic tube with an internal diameter of 1.5 mm was filled with Z100 composite (shade A2, 3M ESPE). Z100 composite was chosen to represent an adhesive resin cement, such as Rely X Unicem (3M ESPE), as both materials are composed of a methacrylate-based monomer reinforced with silanated inorganic fillers (70 wt% for RelyX Unicem and 85 wt% for Z100). Z100 resin composite was used instead of a cement in order to prevent cohesive fractures within the bonded post. The composite filled tube was affixed to the surface of each ceramic specimen and light cured (Elipar

Table 2: *Etching, Silane, and Adhesive Procedure*

Group	Etch Concentration, %	Etching Time, s	Silane Treatment Duration, s	Universal Adhesive Treatment Duration, s
Control	None	—	—	20
5HF	5	20	—	20
9.5HF	9.5	60	—	20
S	None	—	20	20
5HFS	5	20	20	20
9.5HFS	9.5	60	20	20

Abbreviations: 5HF, 5% hydrofluoric acid (HF) etch for 20 s; 9.5HF, 9.5% HF etch for 60 s; S silane with no HF; 5HFS, 5% HF for 20 s + silane; 9.5HFS, 9.5% HF for 60 s + silane.

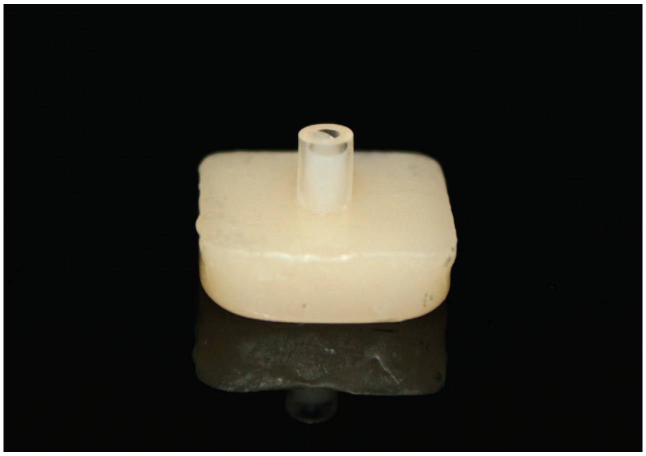

Figure 1. *Composite tube affixed to the coupon of lithium disilicate.*

S10, 3M ESPE) on four sides for 20 seconds per side (Figure 1). After storage in deionized water for 24 hours at 37°C, the specimens were thermocycled for 10,000 cycles (six days) between 5°C and 50°C water baths (15-second dwell time). Prior to shear bond strength testing, the plastic tube was removed to reveal a cylinder of composite. The specimens were mounted into a steel fixture in a universal testing machine (Instron 5565, Canton, MA, USA). A sharpened stylus applied a shear load to the side of the composite cylinder until failure at a crosshead speed of 1 mm/min (Figure 2). The peak failure load and surface area of the composite cylinder were used to calculate the shear bond strength (MPa). An additional specimen from both etching conditions was sputter-coated and imaged under a scanning electron microscope (SEM) for observation of the etching pattern.

A two-way analysis of variance (ANOVA) and Tukey post hoc analysis were used to analyze the differences in the bond strength values based on HF etching and silane treatment (α=0.05).

Table 3: *Bond Strength of Lithium Disilicate to Composite Resin with Different Surface Treatments (Mean ± Standard Deviation [Group Abbreviation])*[a]

	Bond Strength, MPa	
	No Silane Application	**Silane Application**
No HF	1.82 ± 2.0 A (control)	12.55 ± 5.0 A (S)
5% HF	19.08 ± 3.0 B (5HF)	40.47 ± 4.2 B (5HFS)
9.5% HF	24.93 ± 2.6 C (9.5HF)	37.50 ± 5.1 B (9.5HFS)

Abbreviations: 5HF, 5% hydrofluoric acid (HF) etch for 20 s; 9.5HF, 9.5% HF etch for 60 s; S silane with no HF; 5HFS, 5% HF for 20 s + silane; 9.5HFS, 9.5% HF for 60 s + silane.
[a] *Groups in each column with similar capital letters are not statistically different.*

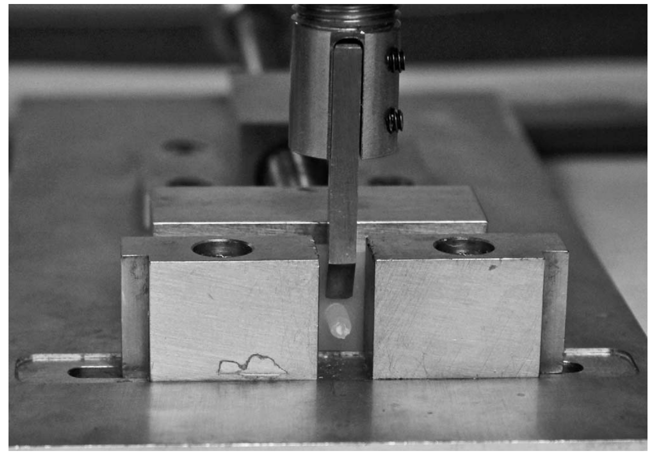

Figure 2. *Lithium disilicate specimen mounted in the universal testing device.*

RESULTS

The means and standard deviations of the bond strength values for each group are presented in Table 3. The two-way ANOVA determined that there were significant differences between HF etching, silane treatment, and the interaction between the HF and silane groups ($p<0.01$). As the factor "silane treatment" was found to be significant, silane treatment was shown to provide higher shear bond strength without HF etching and at both concentrations of HF etching. Individual one-way ANOVA and Tukey post hoc analyses were performed for the silane and no silane groups. Shear bond strength values for each etch concentration were significantly different ($p<0.01$) and could be divided into significantly different groups based on silane treatment: no silane treatment: no HF < 5% HF < 9.5% HF; and RelyX silane treatment: no HF < 5% HF and 9.5% HF. Therefore, the significant interaction could be explained by the fact that without silane treatment, 9.5% HF etching produced higher bond strength than did 5% HF etching, whereas this was not the case if the material was treated with silane. The SEM images of the 5% HF etched lithium disilicate showed the presence of elongated crystals after partial disintegration of the silica matrix (Figures 3A and 3B). The 9.5% HF etched surface showed a more distinct etching pattern with more dissolved matrix (Figures 3C and 3D).

DISCUSSION

The use of HF etching improved resin-lithium disilicate bond strength regardless of whether or not silane was applied. Therefore, we can reject the null hypothesis that HF etching will not affect the

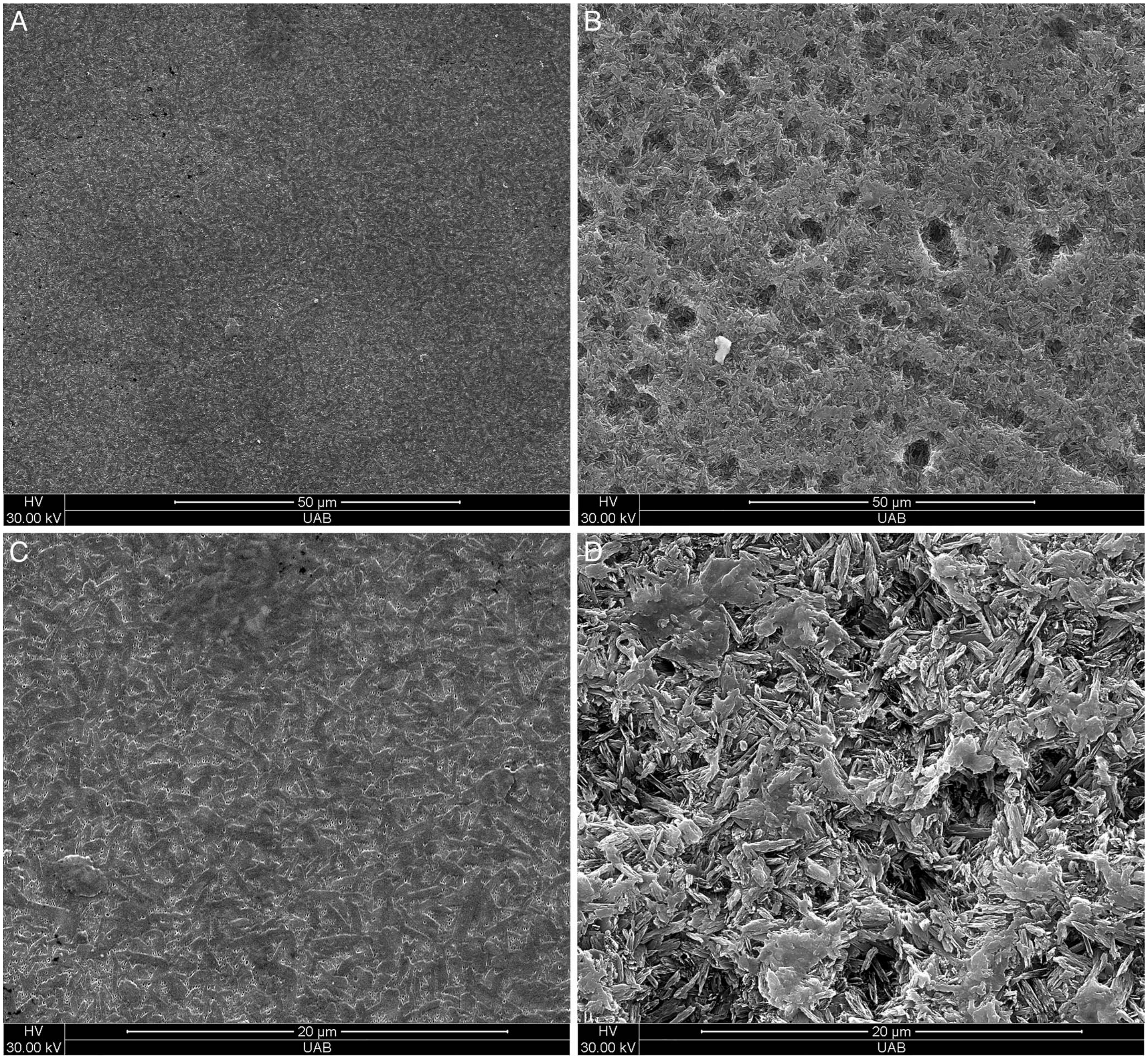

Figure 3. *Etching pattern of lithium disilicate etched for 20 seconds with 5% HF acid at 3000× (A) and 10,000× (B) and for 60 seconds with 9.5% HF acid at 3000× (C) and 10,000× (D).*

bond strength of resin bonded to lithium disilicate pretreated with a universal composite. The concentration/application time of HF etchant did not have a significant effect on bond strength for the specimens that were given a coat of silane. The specimens that were not silanated, however, showed significantly greater bond strength with 60 seconds of 9.5% HF than with 20 seconds of 5% HF. Although the study clearly demonstrates that optimal bonds are achieved by HF etching and silanating, if an additional silane step is not taken prior to applying a universal adhesive, the use of 9.5% HF for 60 seconds can increase bond strength. Perhaps the explanation for this difference is that the specimens that did not receive silane were more dependent on the micromechanical retention provided by the etch pattern in the ceramic. The SEM images show that there is considerably deeper etching pattern present on the specimen etched for 60 seconds at 9.5% HF (Figure 3A-D). Therefore, the nonsilanated specimens could achieve higher bond strengths with a deep etching pattern in the 9.5% HF. The specimens that were silanated, on the other hand, could rely on the chemical bond facilitated by the silane molecules.

The choice to etch for 20 seconds with 5% HF and for 60 seconds with 9.5% HF was based on the manufacturer's recommendations. Previous studies have suggested that etching with 4.9% HF for over 90 seconds[20] or with 9% HF for 120 seconds[21] decreases the strength of the lithium disilicate. As the bond strength in this study did not improve when increasing the etching concentration from 5% HF (20 seconds) to 9.5% HF (60 seconds), it is beneficial to adopt a regimen of 5% HF at 20 seconds. This etching protocol will save the clinician time and preserve the strength of the lithium disilicate.

The results of this study also reveal that silane treatment prior to application of a universal adhesive significantly improved the bond strength regardless of the method of etching. We can therefore reject the null hypothesis that there is no difference in bond strength between resin and lithium disilicate when a silane treatment is applied. This result suggests that the constituent silane in the universal adhesive was not effective in optimizing the ceramic-resin bond. Clinicians should therefore pretreat lithium disilicate with a coat of silane prior to applying the universal adhesive. A study by Panah and others[15] showed that microshear bond strength between lithium disilicate and composite resin improved from 4.10 MPa to 14.58 MPa when silane was applied. Additionally, the microshear bond strength improved from 14.04 MPa to 24.70 MPa when silane was applied to lithium disilicate that had been HF etched.[15] This study further confirms that lithium disilicate should undergo both sufficient etching and silanization prior to bonding.

One limitation of this study is that only one brand of universal adhesive was tested. There are several commercially available universal adhesives that have unique compositions. The specific chemicals and concentrations used in each brand of adhesive may have different interactions with the method of surface pretreatment.

CONCLUSIONS

Optimal bonds are achieved by HF etching and application of silane to lithium disilicate prior to application of a universal adhesive. When using the universal adhesive in conjunction with silane, it is beneficial to adopt a regimen of 5% HF at 20 seconds in order to minimize surface damage to the ceramic while preserving the bond strength. If an additional silane step is not taken prior to applying a universal adhesive, the use of 9.5% HF for 60 seconds can increase bond strength. As the constituent silane and MDP in the universal adhesive were not effective in optimizing the ceramic-resin bond, silane should always be applied to lithium disilicate prior to bonding.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article except for the following: Dr Burgess has, through the University of Alabama at Birmingham, contracts and research grants from the following: GC, 3M ESPE, Ivoclar, Dentsply, Septodont, Glidewell, Discus, VOCO, Shofu, Ultradent, Noritake, and DMG. However, Dr Burgess received no honoraria, nor did he collect any fee, for the submission of this manuscript.

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