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Does a Self-etching Ceramic Primer Improve Bonding to Lithium Disilicate Ceramics? Bond Strengths and FESEM Analyses

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Clinical Relevance

Etching with hydrofluoric acid (HF) followed by a silane coupling agent may still be the most reliable surface treatment for lithium disilicate ceramics. Clinicians may need to be aware of HF etchants that result in surface overetching.

SUMMARY

Objective: To compare the effect of hydrofluoric acid (HF) vs self-etching ceramic primer on resin cement microshear bond strength (μSBS) and ultramorphology of lithium disilicate (LD) ceramic.

Methods and Materials: LD (IPS e.max CAD, Ivoclar Vivadent) blocks (14×4×2 mm^3) were polished to 1200 grit and assigned to nine groups (n=5): CON: control, no LD surface treatment; IVO: 5.0% HF (IPS Ceramic Etching Gel, Ivoclar Vivadent); VIT: 5.0% HF (Vita Ceramics Etch, VITA Zahnfabrik); FGM: 5.0% HF (Condac Porcelana, FGM); ULT: 9.0% HF (Porcelain Etch, Ultradent); PRM: 9.6% HF (Premier Porcelain Etch Gel, Premier); BIS: 9.5% HF (Porcelain Etchant, Bisco Inc); DEN: 10.0% HF (Condicionador de Porcelanas, Dentsply Brazil); and MEP: self-etching ceramic primer (Monobond Etch & Prime, Ivoclar Vivadent). For all HF groups and control, an MDP-containing silane solution (MB$^+$, Monobond Plus, Ivoclar Vivadent) was applied on rinsing the HF gel and air drying. Three transparent matrices for each specimen were filled with light-cured resin cement (Variolink Veneer, Ivoclar Vivadent). After storage in water for 48 hours at 37°C, specimens were tested in shear mode to measure μSBS. Mode of failure was analyzed at 50×. Statistical analysis included one-way analysis of variance and the Duncan *post hoc* test (α=0.05). Thirty-six additional LD specimens were assigned to the same experimental groups (n=4) and observed under a field-emission scanning

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electron microscope (FESEM) at magnifications ranging from 10,000× to 100,000×.

Results: IVO resulted in statistically higher mean μSBS than all the other groups. MEP resulted in statistically lower μSBS than all HF groups. The failure mode for MEP was predominantly adhesive. The most frequent failure mode for the HF groups was mixed. CON resulted in 100% pretesting failures. For FESEM, no retentive pattern was observed for CON specimens. MEP resulted in the least pronounced etching pattern, few areas around crystals exhibited a slight increase in retention pattern compared to the control group. All HF gels created microporosities on the LD surface with distinct etching patterns. VIT and DEN resulted in an LD ultramorphology that suggested overetching.

Conclusions: HF etching followed by a silane solution resulted in higher bond strengths than a self-etching ceramic primer. Some HF gels may cause overetching of the LD intaglio surface.

INTRODUCTION

The creation of microporosities on glass-matrix ceramics using hydrofluoric acid (HF)[1] followed by a silane coupling agent[2] has been the standard procedure for adhesive cementation of porcelain restorations. Bonded glass-matrix ceramic restorations mimic the biomechanical properties, strength, and esthetics of the original tooth[3,4] while performing very well in the clinical setting.[4,5]

Lithium disilicate (LD) glass-matrix ceramic is indicated for full- and partial-coverage bonded ceramic restorations. HF partially dissolves the glass-matrix phase by reacting with silicon dioxide.[6] As a result, HF creates a network of microporosities on the LD surface,[7] forming a microretentive pattern for resin cement interlocking,[8] which leads to higher mean bond strengths compared to nonetched LD.[7-9] Silane coupling agents enhance the resin cement bonding by creating a chemical interaction between the silica in the glass phase of the glass-matrix ceramics and the methacrylate groups of the resin cement.[6,10] 10-Methacryloyloxydecyl dihydrogen phosphate (MDP)– containing silane coupling agents have the potential to interact chemically with LD.[11] The presence of MDP may help maintain the stability of the bonds to LD.[12]

Ivoclar Vivadent, the major manufacturer of LD ceramic, recommends etching the intaglio surface with ≈5% HF gel (IVO, IPS Ceramic Etching Gel, Ivoclar Vivadent, Schaan, Liechtenstein) for 20 seconds. Recently, other ≈5% HF gels have become available, specifically indicated to etch LD. These etchants have specific differences, including gel viscosity, color, packaging type (bottle or syringe), buffering capacity, and the presence of sulfuric acid mixed with HF. However, many of these HF gels have not been tested independently on LD. Therefore, it is relevant to compare their effect on the ultramorphology of LD intaglio surfaces and resulting bond strengths.

A self-etching ceramic primer was introduced in 2015 (MEP, Monobond Etch & Prime, Ivoclar Vivadent). The manufacturer claims that this new primer is capable of etching and priming LD without the need for a separate HF etching step and a separate silane coupling agent. The peer-reviewed literature is scarce regarding the ability of this new all-in-one ceramic primer to promote bonding to LD comparable to that obtained with HF-etched and silane-treated LD. The objective of this study was to compare the effect of HF vs self-etching ceramic primer on the resin cement microshear bond strengths (μSBS) to LD ceramic and respective ultramorphology using field-emission scanning electron microscopy (FESEM). The null hypothesis tested was that the etching protocol would not influence the etching pattern and resin cement μSBS to LD.

METHODS AND MATERIALS

Microshear Bond Strength

Ten IPS e.max CAD blocks LT A2/C14 (Ivoclar Vivadent) were cut into 45 rectangular sections (14×4×2 mm^3) using a slow-speed diamond saw (Model 650, South Bay Tech Inc, San Clemente, CA, USA) under water irrigation. After cleaning ultrasonically with distilled water for 10 minutes, LD specimens were fired following the crystallization program recommended by the manufacturer. The resulting LD specimens were positioned in polyvinyl chloride plastic rings and embedded with epoxy resin (Epo-Thin Resin, Buehler Inc, Lake Buff, IL, USA). The LD surfaces were then ground flat with abrasive silicon carbide paper (360, 600, and 1200 grit) for one minute each under water. An acid-resistant, double-sided adhesive tape (Scotch Permanent Double Sided Tape, 3M, St Paul, MN, USA) was perforated with three 0.8-mm-diameter holes and positioned over the LD surface. Nine experimental groups (n=5) were created (Table 1):

Table 1: *Materials, Manufacturers, Batch Numbers, and Compositions*

Material	Brand Name (Manufacturer), Batch Number	Composition
Lithium disilicate glass ceramic	IPS e.max CAD (Ivoclar Vivadent), S17323	SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, other oxides, coloring oxides
Ceramic etchant	IPS Ceramic Etching Gel (Ivoclar Vivadent), S13497	$\leq$5.0% hydrofluoric acid
Ceramic etchant	Vita Ceramics Etch (VITA Zahnfabrik), 42530	$\leq$5.0% hydrofluoric acid and $\leq$10% sulfuric acid
Ceramic etchant	Condac Porcelana 5% (FGM Produtos Odontológicos), 100915	5.0% hydrofluoric acid
Porcelain etchant	Porcelain Etch (Ultradent Products, Inc), BBHKX	Buffered 9.0% hydrofluoric acid
Porcelain etchant	Premier Porcelain Etch Gel (Premier Dental Products), PE4343-1	9.6% hydrofluoric acid
Porcelain etchant	Porcelain Etchant (Bisco, Inc), 1500002557	Buffered 9.5% hydrofluoric acid
Porcelain etchant	Condicionador de Porcelanas (Dentsply Brasil), 146312H	10.0% hydrofluoric acid
Silane coupling agent	Monobond Plus (Ivoclar Vivadent), U03528	Ethanol, 3-trimethoxysilylpropyl methacrylate, 10-MDP (MDP), sulfide methacrylate
Self-etching ceramic primer	Monobond Etch & Prime (Ivoclar Vivadent), V09349	Butanol, trimethoxypropyl methaycrylate (silane), $\leq$10% tetrabutylammonium dihydrogen trifluoride, methacrylated phosphoric acid ester, colorant
Light-cured resin cement	Variolink Veneer (Ivoclar Vivadent), S20664	Paste of dimethacrylates, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers, pigments

- CON: no LD treatment
- IVO: LD etched with 5.0% HF gel (IPS Ceramic Etching Gel, Ivoclar Vivadent)
- VIT: LD etched with 5.0% HF gel (Vita Ceramics Etch, VITA Zahnfabrik H. Rauter GmbH & Co KG, Bad Säckingen, Germany)
- FGM: LD etched with 5.0% HF gel (Condac Porcelana 5%, FGM Produtos Odontológicos, Joinville, Brazil)
- ULT: LD etched with 9.0% HF gel (Porcelain Etch, Ultradent Products, Inc, South Jordan, UT, USA)
- BIS: LD etched with 9.5% HF gel (Porcelain Etchant, Bisco, Inc, Schaumburg, IL, USA)
- PRM: LD etched with 9.6% HF gel (Premier Porcelain Etch Gel, Premier Dental Products, Plymouth Meeting, PA, USA)
- DEN: LD etched with 10.0% HF gel (Condicionador de Porcelanas, Dentsply Indústria e Comércio Ltda, Petrópolis, Brazil)
- MEP: LD treated with a self-etching ceramic primer (Monobond Etch & Prime, Ivoclar Vivadent)

For the HF groups, a drop of gel was applied directly on the LD surface for 20 seconds. The HF gel was thoroughly rinsed off with water from an air-water syringe for 30 seconds. The LD surface was cleaned ultrasonically in distilled water for 180 seconds and air-dried for 60 seconds. For the HF and control groups, one coat of MB^+ (Monobond Plus, Ivoclar Vivadent) silane coupling agent was applied with a small brush, left on the LD surface for 60 seconds, and then air-dried with a strong jet of water- and oil-free air for 10 seconds. For the MEP group, one drop of the self-etching ceramic primer was scrubbed with a small brush for 20 seconds, left on the LD surface for 40 seconds, and then thoroughly rinsed off with air-water spray and air-dried with a strong jet of water- and oil-free air for approximately 10 seconds.

Subsequently, three polyethylene transparent Tygon tubes (Tygon Medical Tubing Formulations 54-HL, Saint Gobain Performance Plastics, Akron, OH, USA), with an internal diameter of 0.8 mm and a height of 0.5 mm, were positioned over the LD surface in each specimen. A light-cured resin cement (Variolink Veneer, shade Medium Value 0, Ivoclar Vivadent) was carefully packed inside each tube, and a clear Mylar matrix strip was placed over the filled Tygon tube and pressed gently into place. The resin cement was light-cured for 40 seconds using an LED light-curing unit (Bluephase N, Ivoclar Vivadent) with a light energy of 48 J/cm^2.

Specimens were stored in water for 48 hours at 37°C. Each specimen was positioned onto the universal testing machine, and a thin orthodontic wire (0.2-mm diameter) was looped around each resin cement cylinder. The setup was aligned to ensure the accurate orientation of the shear forces.[13] The crosshead speed of a universal testing machine (Instron 4444, Instron Corporation, Canton, MA, USA) was set at 1 mm/min, and the specimens were tested until failure. The μSBS (MPa) was calculated by dividing the load at failure by the surface area (mm^2). After testing, the specimens were examined

under an optical microscope (Leica DM4000 M, Leica Microsystems GmbH, Wetzlar, Germany) in dark-field mode at 50× magnification. The failure mode was classified as cohesive in resin cement (CR, failure exclusively within resin cement), adhesive (A, failure exclusively between the resin cement–LD interface), or mixed (M, failure at the resin cement–LD interface that included any size of cohesive failure of the resin cement over the bonding area).

Statistical Analysis

Statistical analysis was carried out using IBM SPSS 22 (IBM, Armonk, NY, USA) statistical software. The Kolmogorov-Smirnov test showed the sample fit the assumption of normality (p=0.129). The Levene test (p=0.232) demonstrated that the sample variances were not different. Subsequently, a one-way analysis of variance was computed, followed by the Duncan *post hoc* test ($p<0.05$).

FESEM Analysis

Four LD blocks were cut into 36 sections (4×4×1 mm^3) using a slow-speed diamond saw (Model 650, South Bay Tech Inc) under water irrigation. After cleaning ultrasonically with distilled water for 10 minutes, specimens were fired following the crystallization program recommended by the manufacturer.

The specimens were assigned to the same nine groups (n=4), except that silane was not used to prevent masking of the LD surface morphology. Specimens were cleaned ultrasonically with distilled water for 180 seconds, air-dried, and left in a vacuum desiccator for 24 hours. Specimens were mounted on aluminum stubs with adhesive carbon tape (PELCO Carbon Conductive Tape, Ted Pella Inc, Redding, CA, USA) and colloidal quick-drying silver paint (PELCO Colloidal Silver, Ted Pella Inc). Sputter coating was carried out with gold palladium by means of a sputter coater (SCD 500 EVN, Bal-Tec AG, Balzers, Liechtenstein) at 40 mA for 40 seconds. Specimens were observed under a FESEM (JSM-6701F, JEOL, Tokyo, Japan) at an accelerating voltage of 5.0 kV and a working distance of 3.0 to 6.8 mm with at magnifications ranging from 10,000× to 40,000×.

RESULTS

Microshear Bond Strength

Mean μSBS ± SD (MPa) and failure mode are displayed in Table 2. Means with different letters indicate a significant difference ($p<0.05$). All CON specimens and two MEP specimens failed prior to testing and were assigned a value of 0 MPa. Mean μSBS ranged from 0 to 15.0 MPa. IVO resulted in statistically higher mean μSBS (15.0±4.1 MPa) than all the other groups. All other HF groups resulted in similar mean μSBS. However, the HF groups resulted in significantly higher mean μSBS than MEP (3.8±1.9 MPa).

Table 2: *Group, Etchant, Ceramic Primer, Failure Mode (%), and Mean μSBS ± SD. (MPa)*

Group	Etchant	Ceramic Primer	Failure Mode (%)	Mean[a] ± SD
CON	None	Monobond Plus Ivoclar Vivadent	A = 100 M = 00 CR = 0 CC = 0	0.0 ± 0.0 D
IVO	≤5% HF Ivoclar Vivadent		A = 40 M = 47 CR = 13 CC = 0	15.0 ± 4.1 A
VIT	≤5% HF <10% sulfuric acid VITA Zahnfabrik		A = 33 M = 67 CR = 0 CC = 0	8.1 ± 2.7 B
FGM	5% HF FGM Produtos Odontológicos		A = 27 M = 67 CR = 6 CC = 0	7.6 ± 1.7 B
ULT	9% HF Ultradent Products, Inc		A = 34 M = 60 CR = 6 CC = 0	8.0 ± 2.2 B
PRM	9.6% HF Premier Dental Products		A = 14 M = 80 CR = 6 CC = 0	8.5 ± 2.6 B
BIS	9.5% HF Bisco, Inc		A = 27 M = 73 CR = 0 CC = 0	8.6 ± 2.0 B
DEN	10% HF Dentsply Brasil		A = 40 M = 60 CR = 0 CC = 0	8.7 ± 2.8 B
MEP		Monobond Etch & Prime Ivoclar Vivadent	A = 73 M = 27 CR = 0 CC = 0	3.8 ± 1.9 C

[a] *Means with the same letter are not significantly different at* $p < 0.05$.
Abbreviations: A, adhesive; M, mixed; CR, cohesive in resin; CC, cohesive in ceramic.

FESEM Analysis

Figure 1 shows the morphology of untreated LD or CON (Figure 1A), LD treated with HF (IVO [Figure 1B], VIT [Figure 1C], FGM [Figure 1D], ULT [Figure 1E], BIS [Figure 1F], PRM [Figure 1G], DEN [Figure

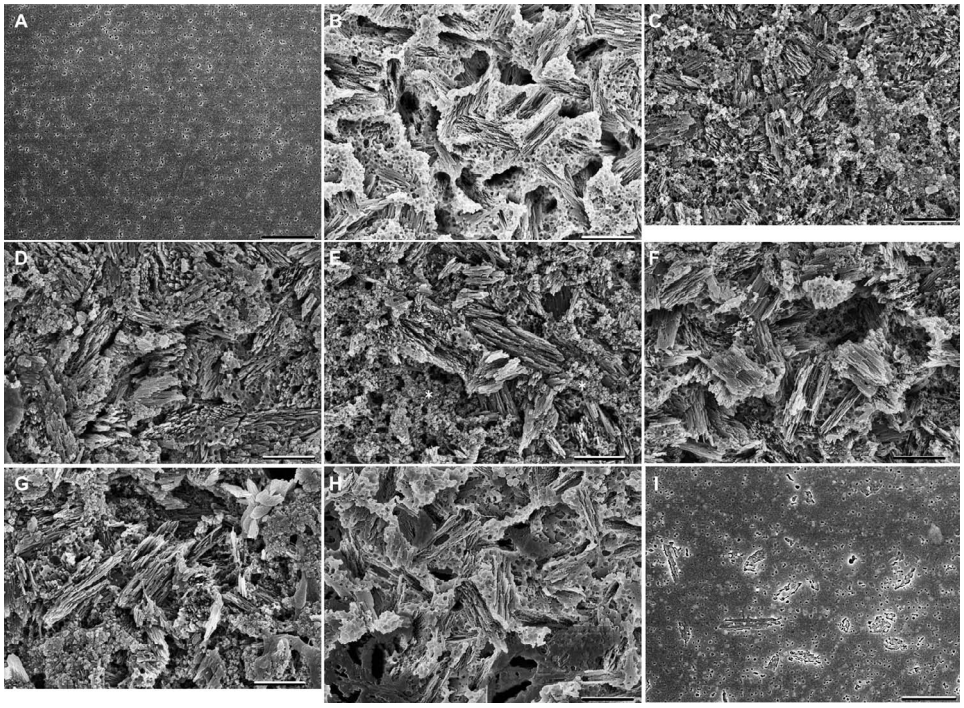

Figure 1. *FESEM micrographs of LD surface after the following treatments: (A): No surface treatment (CON). (B): 5.0% HF (IVO). (C): 5.0% HF (VIT). (D): 5.0% HF (FGM). (E): 9.0% HF (ULT). (F): 9.5% HF (BIS). (G): 9.6% HF (PRM). (H): 10.0% HF (DEN). (I): Self-etching ceramic primer (MEP). Asterisks in Figure 1E denote residual surface deposits. Bar = 1 μm, original magnification = 20,000×.*

1H]), and LD treated with MEP (Figure 1I) at 20,000×. MEP resulted in the least pronounced etching pattern (Figure 1I).

Figure 2 shows a comparison between CON (Figure 2A) and MEP (Figure 2B) at 40,000×. The morphology of LD in the control group showed a smooth surface without retentive features in which 5- to 40-nm-wide nanoporosities were depicted (Figure 2A). In MEP, the LD surface displayed nanoporosities similar to those in the control group but with a larger diameter (20 to 90 nm), while sporadic areas around crystals exhibited a slight increase in retention pattern (Figure 2B) compared to the control group when observed at high magnification (Figure 2A). Comparing the FESEM morphology of the polished LD surfaces (CON) with that of MEP-treated LD surfaces, the ultrasonic cleaning for 180 seconds was able to remove any residual monomer that might have been left by the self-etching ceramic primer MEP (Figure 2B).

Figure 3 shows the morphology of LD treated with VIT (Figure 3A) and FGM (Figure 3B). Figure 3C depicts a high magnification of LD treated with IVO. Figure 4 shows higher magnifications of the morphology of LD treated with BIS (Figure 4A) and with DEN (Figure 4B).

LD specimens treated with IVO (Figure 1B) and FGM (Figure 1D) displayed a more defined etching pattern when compared with ULT (Figure 1E), which displayed the least pronounced etching pattern among the HFs tested, with residual surface deposits resembling a precipitate (Figure 1E). Additionally, IVO resulted in the most consistent and homogeneous etching pattern with an array of

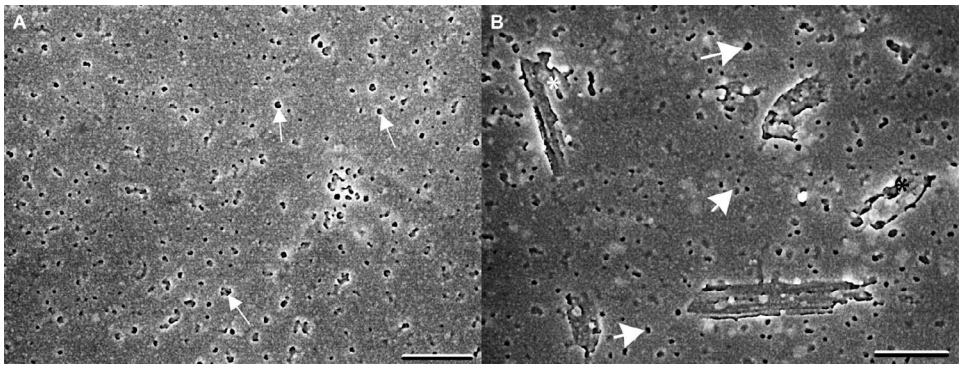

Figure 2. *FESEM micrographs of LD surface (A) without surface treatment (CON) and (B) after treatment with self-etching ceramic primer (MEP). Arrows in Figure 2A show 5- to 40-nm-wide nanoporosities. Arrows in Figure 2B show 20- to 90-nm-wide nanoporosities; asterisks show sporadic retention areas around crystals. Bar = 0.5 μm, original magnification = 40,000×.*

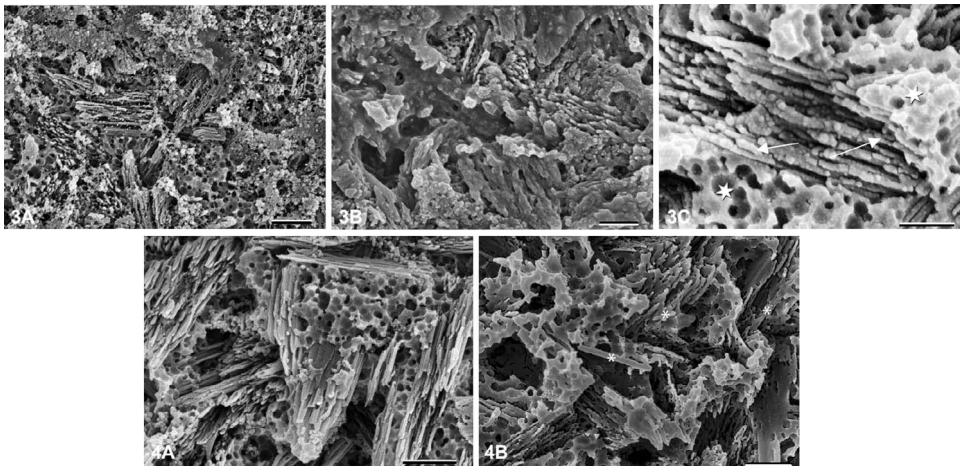

Figure 3. *FESEM micrographs of LD surface after (A) 5% HF (VIT) and (B) 5% HF (FGM). Bar = 0.5 μm, original magnification = 30,000×. (C) Higher magnification of micrograph in Figure 1B (IVO). The glass phase has a honeycomb-like morphology (stars). Arrows show LD crystals attached to the glass phase. Bar = 0.2 μm, original magnification = 100,000×.*

Figure 4. *FESEM micrographs of LD surface after the following treatments: (A): 9.5% HF (BIS). (B): 10.0% HF (DEN). Arrows in Figure 4A and 4B show areas resembling overetching. Pointer in Figure 4B shows unsupported LD crystals. Asterisks in Figure 4B show areas of the glass phase that were detached from the ceramic core, while loose crystals displayed a smoother surface texture. Bar = 0.5 μm, original magnification = 40,000×.*

exposed crystals without apparent overetched areas (Figure 1B). LD crystals were attached to the glass phase, and the latter showed a honeycomb-like morphologic characteristic at 100,000× (Figure 3C). One LD specimen treated with FGM showed areas (Figure 3B) without the same homogeneous etching pattern as seen in Figure 1D. Figure 3B also shows an area where the honeycomb-like glass phase ceramic pattern was not as clear as seen in Figure 1D.

VIT showed areas resembling overetching (Figure 3A). An intermediate deep etching pattern was obtained with BIS (Figures 1F and 4A) and PRM (Figure 1G). DEN specimens displayed the most pronounced etching pattern with unsupported LD crystals and areas with morphology compatible with overetching (Figure 1H). Additionally, areas of the glass phase were detached from the ceramic core, while loose crystals displayed a smoother surface texture at 100,000× (Figure 4B).

DISCUSSION

The null hypothesis was rejected. The "all-in-one" self-etching ceramic primer tested (MEP) resulted in statistically lower mean μSBS to LD than any of the HF etchants. MEP also resulted in the least pronounced etching pattern compared to the groups in which HF was used, which may preclude a durable micromechanical bonding.

MEP contains tetrabutylammonium dihydrogen trifluoride, trimethoxypropyl methaycrylate (silane), and methacrylated phosphoric acid ester.[14] Several sources of fluoride have been investigated for ceramic etching, including acidulated phosphate fluoride, titanium tetrafluoride, and ammonium bifluoride.[15-20] Tetrabutylammonium dihydrogen trifluoride in MEP, also a source of fluoride, is based on ammonium bifluoride (ABF), which is less toxic[21] and less hazardous than HF.[16] It has been reported that the etching patterns of ABF are very similar of those created when HF is applied for a shorter time and at a lower concentration. Therefore, the interaction of ammonium fluoride–based porcelain etchants with ceramics may be similar to that of a low concentration of HF acid applied for a short period.[6] Although ABF was more effective on Dicor castable glass ceramic (DICOR, Dentsply International, York, PA, USA) than HF,[16] etching current glass-matrix ceramics with HF results in statistically higher mean tensile bond strengths compared to etching with ABF.[19] This is in agreement with the results of our study, as MEP contains ammonium fluoride.

Recent studies have reported that mean μSBS were statistically similar when 5% HF was compared with MEP.[22,23] Conflicting results may be explained by differences in testing methodology. For example, the present study used LD polished up to 1200-grit silicon carbide paper for μSBS and FESEM analysis. A pilot study in our laboratory revealed that HF etches glass-matrix ceramic deeper with residual crevices remaining on the surface when specimens are not polished to fine-grit sandpaper. While the LD intaglio surface may be rougher in a clinical situation than in laboratory studies, polished LD is used in the laboratory for standardization purposes.[24-29] Highly polished LD may result in more

consistent ceramic ultramorphology, making it possible to compare the LD etching pattern among different experimental groups. The ultramorphology of LD milled with a standard bur and treated with HF or with MEP was recently evaluated.[30] MEP resulted in a slight increase in micromechanical retention only in the groove areas created during the milling process.[30] As a result of this minor morphological difference, the mean μSBS to milled LD might be slightly higher than that obtained to polished LD. However, the milling process results in a less uniform surface, as it is not possible to standardize the LD surface. This lack of standardization may cause a wider standard deviation of bond strengths for milled surfaces.

In the present study, 10% HF (DEN) specimens displayed the most pronounced etching pattern with unsupported crystals and areas with morphology compatible with overetching. Similarly, VIT showed areas resembling overetching, which may be a result of the presence of <10% sulfuric acid in the composition of VIT.[31] Other studies have reported potential overetching with other HF etchants, especially when used for prolonged etching times. A study using scanning electron microscopy/atomic force microscopy showed that etching with 9.6% HF gel resulted in preferential dissolution of the glass matrix, but the extent of LD surface etching depth increased with etching time.[25] Partially supported crystals within the glass matrix were lost with an etching time longer than 20 seconds. LD became progressively more irregular with numerous voids forming with increasing HF etching time.[25] Another study reported that LD etched with 9.0% HF gel (ULT) for 120 seconds resulted in lower flexural strength than unetched LD,[28] which suggests that overetching LD may weaken the restoration. Xiaoping and others[29] observed that etching LD with 9.5% HF (BIS) for 120 seconds dissolved much of the glass matrix, causing unsupported crystals and an increase in the number of microdefects. Zogheid and others[32] reported that etching LD with 4.9% HF (IVO) for 20 seconds did not result in a significant reduction of flexural strength compared to that of unetched LD. However, etching LD for 90 or 180 seconds with IVO resulted in a significantly lower mean flexural strength compared to that of LD etched for 20 seconds.[32]

Mean μSBS to HF-etched and silane-coated LD using light-cured resin cement obtained in our study are in agreement with other authors.[33,34] Recently, Perdigão and others[33] measured a mean μSBS of 14.7 MPa to HF-etched and MDP-containing silane (MB^+)-coated LD using a light-cured resin cement. Using 10% HF (DEN) to HF-etched and MDP-free silane-coated LD, Baratto and others[34] reported a mean μSBS of 12.5 MPa also using a light-cured resin cement. The light-cured resin cement resulted in lower μSBS than the dual-cure resin cement used in the same study.[34] Lise and others[8] used dual-cure resin cements (Variolink II; Multilink Automix, Ivoclar Vivadent; RelyX Unicem 2, 3M ESPE, St Paul, MN, USA) applied to LD etched with IVO and coated with MB^+. These authors obtained a mean bond strength greater than 40 MPa,[8] most likely as a result of the higher flexural strength and elastic modulus of dual-cure resin cements when light cured.[35] Variolink Veneer (Ivoclar Vivadent) is a microfilled resin cement[33] with a modulus of elasticity of 4.4 ± 0.4 GPa,[36] while RelyX Unicem 2 (3M ESPE) and Variolink II (Ivoclar Vivadent), both dual-cured resin cements, have a mean modulus of elasticity of 10.5 ± 0.1 GPa[37] and 11 ± 0.5 GPa,[36] respectively.

Kalavacharla and others[38] used 5% HF (IVO) for 20 seconds or 9.5% HF (BIS) for 60 seconds on LD surfaces. Mean bond strengths were not significantly different. However, on analysis under the FESEM, LD etched with 5% HF for 20 seconds displayed elongated crystals after partial disintegration of the silica matrix, while LD etched with 9.5% HF for 60 seconds showed a more distinct etching pattern with wider areas of dissolved matrix.[38] These authors concluded that LD etched with 5% HF for 20 seconds is the ideal etching protocol to minimize surface damage to LD. These findings corroborate in part the results in the present study. IVO resulted in higher mean μSBS than the other HF gels tested in spite of apparent similar etching patterns between IVO and FGM. It is unclear whether the etching depth might have played a role in the magnitude of bond strengths.

MB^+ contains MDP, which has potential for chemical interaction with LD substrates.[11] However, the role of MDP in adhesion to LD is not clear. An MDP-containing silane resulted in higher resin cement bond strength to LD than MDP-containing universal adhesives after 150 days in water and 37,500 thermal cycles.[12] Using this same aging process, Elsayed and others[39] showed that bond strengths of resin cement to LD etched with 5% HF followed by the application of MB^+ were more stable compared to those of universal adhesives. However, a silane without MDP (Calibra Silane, Dentsply) followed by a simplified adhesive (Prime & Bond NT, Dentsply) resulted in similar bond strengths to LD

after aging compared to MB^+.[39] In the present study, MB^+ applied to CON resulted in 100% pretesting failures. Oh and Shen[26] also reported a mean bond strength of 0 MPa to both 1200-grit-polished LD and air-abraded LD without silane application. Mean shear bond strengths to 600-grit-polished porcelain blocks was 0 MPa without HF etching and silane application.[40] Even for specimens that were roughened with a diamond bur, Duzyol and others[41] obtained 100% pretesting failures to nonetched LD. The benefit of the presence of MDP in the composition of the silane coupling agent may depend on the depth of exposure of the glass phase by HF etching.

CONCLUSIONS

Within the limitations this study, the following conclusions can be made:

- HF etching followed by a silane/MDP solution may be more reliable for adhesion to LD than a self-etching ceramic primer.
- The absence of an etching pattern that resulted from the application of MEP may preclude micromechanical bonding.
- The ultramorphology of etched LD surfaces depends on the specific HF gel used. While ≈9% HF should be used with caution, 10% HF should be avoided, as it results in overetching. For HF gels with concentration of ≈5%, VIT is the most aggressive.

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Conflict of Interest

The authors of this article 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.

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