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Surface Treatment and Cementation of Lithium Silicate Ceramics Containing ZrO_2

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CLINICAL RELEVANCE

Hydrofluoric acid followed by silanization or Monobond Etch & Prime is an efficient option for the cementation of lithium silicate and lithium disilicate glass ceramics.

SUMMARY

Objective: To evaluate the effect of different surface treatments on the shear bond strength (SBS) of lithium silicate (LS) and lithium disilicate (LD) ceramics, after thermocycling.

Methods and Materials: For SBS test, 72 ceramic blocks (18×14×2 mm) were made (24 blocks from each ceramic material): VITA Suprinity (LSS), Celtra Duo (LSC), and Lithium disilicate (LD). The blocks were polished with sandpaper of increasing grit (#280, #400, #800, and #1200) and embedded in chemically activated acrylic resin. Afterwards, they were randomly divided into 12 groups (6 blocks per group) according to: "Ceramic" (LD, LSC, and LSS) and "Surface treatment" (HFS: hydrofluoric acid + silane; MEP: Monobond Etch & Prime/Ivoclar). From each treated surface ceramic block, four dual-curing resin cement cylinders (RelyX U200, 3M Oral Care) were prepared using a Tygon tube (Ø=3 mm and h=2 mm) and light cured for 40 seconds (1000 mW/cm²) (N=288/n=24). All specimens were submitted

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to thermocycling (10,000 cycles, 5°C and 55°C, 30 seconds) and then to SBS test at a crosshead speed of 1 mm/min using a 50-kgf load cell. Forty-five additional blocks were made for roughness and SEM analysis. Failure mode was also performed. The data (MPa) were statistically analyzed by one-way analysis of variance (ANOVA), Tukey test (5%), and Weibull analysis. The R_a was analyzed by Kruskal–Wallis and Dunn Test (5%). The other variables were analyzed qualitatively.

Results: ANOVA revealed that "surface treatment" was significant for all ceramic materials ($p<0.05$). The LD-HFS (18.66±3.49), LSC-HFS (16.81±2.62), and LSS-HFS (16.33±3.08) groups had significantly higher SBS than the LD-MEP (7.00±4.2), LSC-MEP (14.12±3.51), and LSS-MEP (13.87±2.52) groups. Complete adhesive failures at the cement–dentin interface were more frequent. Weibull modulus was superior for the LD-HFS (6.22), LSC-HFS (8.8), and LSS-HFS (7.4) groups.

Conclusion: HF followed by silanization is the most suitable surface treatment for the cementation of LS and LD glass ceramics.

INTRODUCTION

Lithium silicate (LS) ceramics are presented, within the class of glass ceramics, for making indirect restorations, such as inlays,[1-3] onlays,[2,3] overlays, and crowns,[2-4] and according to manufacturers, provide a lower probability of fracture compared to conventional glass ceramics, and are also associated with high aesthetics.[3,5,6] Commercially, two types of LS ceramic materials are available, Vita Suprinity (Vita Zahnfabrik—Bad Sackingen, Germany) and Celtra DUO (Dentsply—Hanau Wolfgang, Germany), both of which are mainly made up of submicrometric lithium metasilicate (Li_2SO_3) crystals and orthophosphate nanometer lithium (Li_3PO_4) embedded in a glassy matrix with highly dispersed zirconium dioxide (ZrO_2) (±10 wt%).[2] The main difference between the two materials is the size of the Li_2SO_3 crystals (Li_2SO_3 phase), which appears to be larger in the Celtra Duo (up to 1 μm in length) than in the Suprinity (~0.5 μm).[2,7] Although it is reported that these ceramics are reinforced by zirconia,[5,6,8] it is not in its crystallized form, thus they are considered only glassy ceramics based on LS.[2]

Lithium disilicate (LD) ceramics, on the other hand, are composed in their crystallized phase by crystals of LD embedded in a glassy matrix and have a higher percentage of crystalline phase content (70%) compared to LS (40%-50%).[7] Despite the microstructural differences between these two types of ceramics, some studies report high clinical success rates, both for LS ceramics, reaching 98% for crowns or inlays after 3 years of follow-up,[9] and for LD ceramics that vary from 94.8% after 8 years[10] to 83.5% after 10 years.[11] However, failures in restorations such as fractures and debonding are still common regardless of the type of material, especially in clinical situations in which the substrate does not offer mechanical retention; also the cementation technique has a fundamental role in the clinical longevity of these indirect restorations.[9]

With regard to adhesion, *in vitro* studies have investigated different protocols for the surface treatment of these ceramics.[5,12] Among surface treatments, hydrofluoric acid etching (HF) followed by silanization has been proposed as the ideal treatment for all-glass ceramics.[13,14] However, some studies have reported that this protocol has some disadvantages, such as the high toxicity of HF,[5,15-17] different acid concentrations and variations in conditioning time between materials, besides difficulty in controlling the restoration exposure to acid, which can lead to overconditioning of the piece, most of the time decreasing the mechanical resistance of restorations[7,17-19] or impaired adhesion due to excessive glassy phase dissolution in some materials.[20,21] Thus, surface treatment alternatives to HF have also been investigated, such as airborne-particle abrasion with aluminum oxide (Al_2O_3),[5,22] silicatization associated with silanization,[5,23,24] or self-etching ceramic primer, such as Monobond Etch & Prime (MEP, Ivoclar Vivadent).[17,25]

A recent clinical option for conditioning and silanization in glass ceramic restorations is the self-etching ceramic primer (MEP). Because it contains in its single-bottle composition an aqueous acidic solution of ammonium polyfluoride and silane methacrylate, this primer allows the etching and silanization in a single step.[26] According to studies, this primer decreases the probability of excessive degradation of the silica glass matrix, and the toxic effect of HF, as well as presents a satisfactory clinical performance[17,27] and clinically stable adhesion.[28]

Despite several studies investigating glass ceramic surface treatments, there are still few studies that have used other surface treatments as an option to the use of HF, especially with MEP in LS ceramics. Associated with this, most studies, when investigating these protocols, did not use aging of the adhesive interface through thermocycling,[5,29] which has great clinical implications in long-term adhesion. Therefore, this study aimed to evaluate the effect of different types of surface treatments on the shear bond strength (SBS) of LS and LD ceramics to resin cement after thermocycling. The hypotheses tested were: 1) the type of surface treatment does not influence the bond strength regardless of the type of ceramic; 2) the surface treatment influences the surface roughness, regardless of the type of ceramic.

METHODS AND MATERIALS

The materials used in this study, as well as their respective trademarks, manufacturers, and batches, are shown in Table 1. The flowchart of the design of this research is shown in Figure 1.

Production of Specimens

Computer-aided design—Computer-aided manufacturing (CAD–CAM) blocks of three ceramics (18×14×12 mm): Celtra Duo (LSC) (Dentsply—Hanau-Wolfgang, Hesse, Germany), VITA Suprinity (LSS) (Vita Zahnfabrik—Bad Sackingen, Baden-Württemberg, Germany), and IPS e.max CAD (LD) (Ivoclar Vivadent, Schaan, Liechtenstein) were sectioned on a precision saw (Isso Met 1000 Precision Saw, Buehler, Lake Buff - IL, USA) under constant irrigation, using Extec High Concentration Diamond Wafering Blades (Extec, Enfield, CT, USA), in smaller rectangular blocks (18×14×2 mm) for a total of 63 rectangular blocks (ISO/TS 11405). These blocks were polished with SiC abrasive papers (#280, #400, #800, and #1200, Norton Saint-Gobain, São Paulo, Brazil) and sintered according to the recommendation of each manufacturer in a specific oven. Thirty-six of these blocks were used for the SBS test and 27 blocks (9 blocks of each material) were used for Optical profilometry and Scanning Electronic Microscopy (SEM).

Preparation of Blocks

The blocks were embedded in chemically activated acrylic resin (Classic, São Paulo, Brazil), in a PVC tube (¾ inch). The exposed surface was covered with double-sided tape in order to avoid the covering of this surface by the resin.

After polymerization, all the blocks were ultrasonically cleaned with distilled water for 10 minutes (Vitasonic

Table 1: *Trademarks, Type of Material, Composition, Manufacturers, and Batch Numbers of Products Used in the Study*

Trademark	Type of Material	Composition	Manufacturer	Batch
Celtra DUO HT	Lithium silicate	Fully sintered lithium silicate/phosphate (LSP) glass-ceramic (SiO_2, P_2O_5, Al_2O_3, Li_2O, K_2O, ZrO_2, CeO_2, Na_2O, Tb_4O_7, V_2O_5, Pr_6O_{11}, Cr, Cu, Fe, Mg, Mn, Si, Zn, Ti, Zr, Al).	Dentsply, Hanau Wolfgang, Germany	18031266
Vita Suprinity HT	Lithium silicate	Lithium silicate glass-ceramic (SiO_2, Li_2O, K_2O, P_2O_5, Al_2O_3, ZrO_2, CeO_2)	Vita Zahnfabrik, Bad Säckingen, Germany	40020
IPS e.max CAD HT	Lithium disilicate	SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO and other oxides	Ivoclar Vivadent, Schaan, Liechtenstein	W04573
RelyX Ceramic Primer	Silane	Ethyl alcohol, water, methacryloxypropyltrimethoxysilane	3M, St Paul, MN, USA	N822741
Porcelain Etch 9%	9% Hydrofluoric acid	Hydrofluoric acid, water, thickener, surfactant, coloring	Ultradent, South Jordan, UT, USA	18005525512
Monobond Etch & Prime	Self-etching ceramic primer	Butanol, tetrabutylammonium dihydrogen trifluoride, methacrylated phosphoric acid ester, trimethoxypropyl methacrylate monomer	Ivoclar Vivadent, AG, Schaan, Liechtenstein	V50443
Rely X U200	Self-adhesive resin cement	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers; Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers	3M, St. Paul, MN, USA	660958

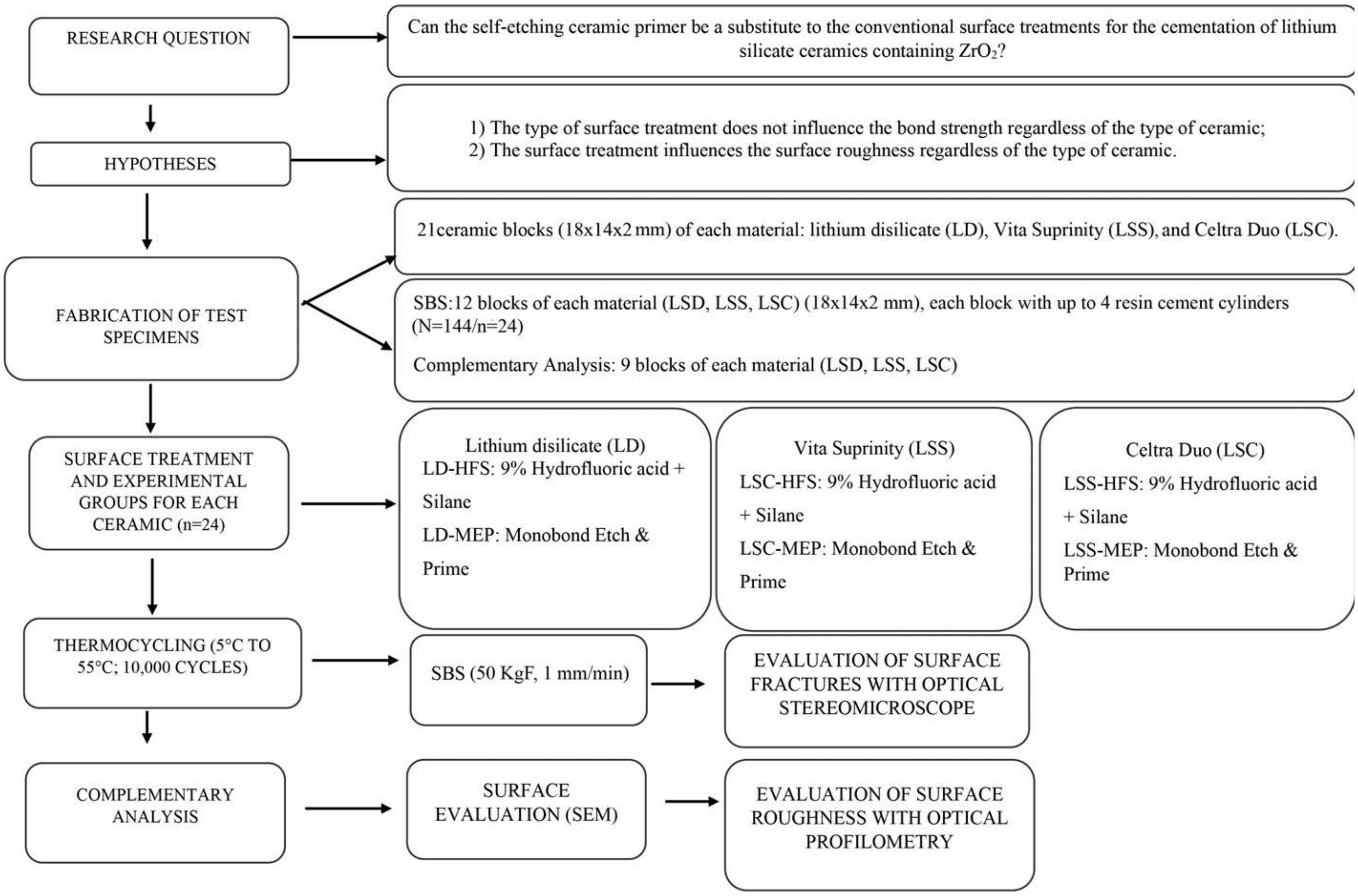

Figure 1. *Flowchart of study protocol. SBS: Shear Bond Strength. Abbreviations: TC, thermocycling; SEM, scanning electronic microscopy; LD, lithium disilicate; LSS, Vita Suprinity; LSC, Celtra Duo; SBS: Shear Bond Strength.*

II, Vita Zahnfabrik – Bad Sackingen, Baden-Württemberg, Germany) and divided into six groups (6 blocks per group). On each ceramic block, four resin cement cylinders were built-up to complete the 24 cylinders per group (N=144/n=24). The groups were divided according to: "glass ceramic" (LSS, LSC, LD) and "surface treatment" HFS: 9% Hydrofluoric acid (Ultradent—South Jordan, UT, USA) + RelyX Ceramic primer (3M Oral Care - St. Paul, MN, USA); MEP: Monobond Etch & Prime (Ivoclar Vivadent—Schaan, Liechtenstein).

Surface Treatments

Prior to surface treatments, all specimens were washed in an ultrasonic bath (Vitasonic II, Vita Zahnfabrik) in 99.8% isopropyl alcohol (Pharmacy of Homeopathy and Manipulation - Juiz de Fora - MG - Brazil) for 8 minutes. The surface treatments of the specimens were carried out by a single operator, on the entire surface of the block, and occurred according to the groups shown in Figure 1 as follows:

- 9% hydrofluoric acid (HFS): 9% Hydrofluoric acid was applied to the surface for 20 seconds for all ceramics, and then washed with air and water spray for 40 seconds, as instructed by the manufacturer. Then, a layer of RelyX Ceramic Primer was applied with the aid of a microbrush (Vigodent, Rio de Janeiro, RJ, Brazil), according to the manufacturer's recommendations.
- Monobond Etch & Prime (MEP): Monobond Etch & Prime was applied for 20 seconds by active friction with a microbrush, waiting 40 seconds for the material action, and then washed with air and water spray for 60 seconds. Finally, they were air dried until the moisture was eliminated. Due to the properties of this conditioning agent and the manufacturer's recommendations, no silane was applied afterwards.

Adhesive Cementation

After the surface treatment, up to four resin cement cylinders (n=24) (RelyX U200 (3M Oral Care) (Ø=3.0

mm, height: 2 mm) were made on the surfaces of the treated ceramics. For the standardization of the diameter of the adhesive area and the height of the cement increment, a matrix of flexible silicone tubing (Tygon tubing, Saint-Gobain Performance Plastic, Miami Lakes, FL)[30] was used. To fix the Tygon, a layer of wax n^{0}7 (New wax, Technew, Rio de Janeiro, Rio de Janeiro, Brazil) was used with the aid of an electric dripper (Plaster, Caxias do Sul—RS, Brazil). Afterward, each resin cement cylinder was individually light cured for 40 sec (VALO/Ultradent) in the standard mode power energy (1000 mW/cm^2). The power of the light–curing unit was measured by a single operator with a radiometer for LEDs (LED Radiometer–Kondortech, São Carlos, SP, Brazil). After photoactivation of each specimen, the Tygon was gently removed after 8-10 minutes, using a #15 scalpel blade, and then each specimen was light cured again, following the same previous protocol.

Thermocycling (TC) and Shear Bond Strength Test (SBS)

All specimens were subjected to thermal aging by means of a thermal cycler (521-D—Ethik Tecnology/Nova Ética—Vargem Grande Paulista—SP) with 10,000 cycles in distilled water at 55°C and 5°C for 30 seconds each, with an exchange interval of 5 seconds. Afterwards, the specimens were submitted to the mechanical shear test (SBS), in a universal testing machine (EMIC—Instron, São José dos Pinhais, PR, Brazil). The load was applied at the base of the cylinder on the adhesive interface using an orthodontic wire (0.4-mm diameter) at a speed of 1 mm/minute and load cell of 50 kgf until fracture of the specimen. The adhesive strength was calculated using the formula: $R = F/A$, where R = Adhesive strength (MPa); F = Force (N); A = Interfacial area (mm). The adhesive area of each block was defined by the area of a circle, calculated by the following formula: $A = \varpi r^2$, where $\varpi = 3.14$ and $r = 1.5$ mm.

Failure Analysis

After SBS testing, failure pattern analysis was performed on all specimens with a stereomicroscope (Carl Zeiss—Oberkochen, Germany) at 40× magnification, determining fractures classified as follows: A) Adhesive in ceramic–resin cement interface; C1) cohesive in ceramic; C2) cohesive in resin cement; mixed 1 (M1) adhesive in ceramic–resin cement interface + cohesive in resin cement; mixed 2 (M2): In ceramic–resin cement interface + cohesive in ceramic.

Optical Profilometry

Twenty-seven additional ceramic blocks, nine of each material (LSS, LSC, and LD), were prepared as previously described and subjected *to* the following surface treatments (n=3): HF, MEP, and control group (no treatment). Subsequently, the blocks were examined using a digital optical profilometer (Wyko, NT 1100, Veeco—Tucson, USA), connected to a computer with imaging software (Vision 32, Veeco—Tucson, USA) for obtaining 20× surface micrographs [qualitative analysis of three-dimensional (3D) geometry], and measurement of surface roughness (R_a). Five readings were performed on each specimen and an arithmetic mean (R_a) of the surface roughness was obtained using the proper system software.

Scanning Electron Microscopy (SEM)

The same surface specimens were examined at 2500× magnification in a TESCAN Scanning Electron Microscope (MEV-FEG, Model MIRA 3, Kohoutovice, Czech Republic) in high vacuum with the aid of a secondary electron Everhart–Thornley detector (ETD).

Statistical Analysis

The sample power calculation performed in this study used the mean and standard deviation of the groups and for this reason, it was performed after the SBS test. The following data were inserted for this calculation: confidence interval: 95%, the mean and standard deviation of the group that presented the higher mean, the mean and standard deviation of the group that presented the lower mean, and the number of tested specimens by group (Table 2). The data obtained from SBS were submitted to the statistical model of analysis of variance, after considering the distribution of residues (Levene test) using the Minitab software (Minitab, version 17, 2013). The residual values, resulting from the adjustment of the adopted model, were examined to assess the suitability of the model for valid statistical inferences, and it was determined that the original data provide an adequate adjustment when they adjusted to a normal probability distribution ($p>0.05$).

Analysis of variance (ANOVA—one way) and Tukey test (5%) were performed for the SBS test for each ceramic individually using the Statistix software (Analytical Software Inc., version 8.0, 2003). Shapiro–Wilk test was used to verify the normality of numerical roughness data, resulting in a nonparametric distribution ($p<0.05$). For these data, the Kruskal–Wallis test and Dunn multiple comparison test were performed using the GraphPad Prism software (GraphPad Software, San Diego, CA, USA). The probability value $p<0.05$ was considered as statistically significant. The failure analysis, SEM, EDS of the surface treatments were carried out through qualitative descriptive analyses.

Weibull analysis was performed to evaluate the

Table 2: *Number (N) and Percentage (%) of Pretest Failure (PTF) During Thermal Aging, Total Number of Specimens Submitted to the Shear Test and Failure Mode (%) of the Groups After SBS Test*

Ceramic	Surface Treatment	Groups	Number of Specimens	Number and Percentage of Spontaneous PTF During Aging	Number and Percentage of Tested Specimens	Percentage by Failure Mode					
						A	C1	C2	M1	M2	Total
Lithium Disilicate (e.max CAD)	HF+Silane	LD-HFS	24	0(0%)	24(100%)	100	—	—	—	—	100%
	MEP	LD-MEP	24	17(70.84%)	7(29.16%)	100	—	—	—	—	100%
Lithium Silicate (Suprinity)	HF+Silane	LSS-HFS	24	0(0%)	24(100%)	12.5	—	—	—	87.5	100%
	MEP	LSS-MEP	24	2 (8.33%)	22 (91.66%)	70.8	—	—	—	29.1	100%
LithiumSilicate (Celtra Duo)	HF+Silane	LSC-HFS	24	0(0%)	24 (100%)	12.5	—	—	—	87.5	100%
	MEP	LSC-MEP	24	5(20.83%)	19 (79.16%)	70.8	—	—	—	29.1	100%

Abbreviations: LD, Lithium disilicate; LSS, Vita Suprinity; LSC, Celtra Duo; HFS, 9% Hydrofluoric acid + Silane RelyX Ceramic primer; MEP, Monobond Etch & Prime; A, Adhesive in ceramic–resin cement interface; C1, Cohesive in ceramic; C2, Cohesive in resin cement; M1, Mixed 1, adhesive in ceramic–resin cement interface + cohesive in resin cement; M2, Mixed 2, adhesive cement–ceramic + cohesive ceramic.

reliability of the SBS, with the Weibull parameter (*m*) and the characteristic strength (σ_0), with a confidence interval of 95%, determined in a $\ln\sigma_c - \ln[\ln 1/(1-F(\sigma_c)]$ diagram (according to ENV 843-5):

$$\ln\ln\left(\frac{1}{1\text{-}F(\sigma_c)}\right) = m\ln\sigma_c - m\ln\sigma_o$$

The characteristic strength is the strength at a failure probability of approximately 63.3%, and the Weibull modulus *m* is used as a measure of the strength distribution, which expresses the structural homogeneity of the material. Statistical analysis was performed using Minitab software (version 17, 2013, Minitab, State College, PA). The level of significance was 5%.

RESULTS

Levene test was performed, and there was no significant difference amongst the standard deviations ($p>0.05$). These results report that the data follow a normal distribution. The sample power calculation was performed by comparing two averages, in which a sample power of 100% was obtained for all ceramics (LD, LSC, and LSS).

Shear Bond Strength Test (SBS)

Table 2 shows a higher number of pretest failures for ceramic: LD-MEP (70%). The means and standard deviations for SBS and Weibull modulus for each material and the comparison among experimental groups are shown in Table 3.

Lithium Disilicate (LD)—ANOVA revealed that the factor "surface treatment" ($p<0.0001$) significantly influenced SBS for LD. When comparing surface treatments, the HFS group (18.66±3.49 MPa) showed significantly greater bond strength.

The Weibull modulus (*m*) and characteristic strength (σ_0) of LD groups were not significant ($p=0.05$). The HFS showed statistical similarity from MEP. Weibull distributions are shown in Table 3 and Figure 2.

Vita Suprinity (LSS)—Regarding LLS, ANOVA revealed that the "surface treatment" ($p=0.004$) was statistically significant. When comparing the surface treatments, Tukey test (5%) revealed that the HFS group (16.33±3.08 MPa) also had a significantly higher mean than the MEP (13.87±2.52 MPa).

The Weibull modulus (*m*) of LSS groups was not significant ($p=0.3$). The characteristic strength (σ_0) of groups were significant ($p=0.001$). The HFS showed higher σ_0 and was statistically different from MEP. The HFS group also had a higher *m* but showed a statistical similarity from MEP. Weibull distributions are shown in Table 3 and Figure 3.

Celtra Duo (LSC)—For LSC, ANOVA revealed that the "surface treatment" ($p<0.006$) was also significant. In the comparison between groups, the HFS (16.81±2.62 MPa) had the highest mean of SBS and was statistically different in relation to the MEP groups (14.12±3.51 MPa) (Tukey test $p<0.05$).

The Weibull modulus (*m*) of LSC groups were significant ($p=0.006$). The HFS showed the highest (*m*) and was statistically different from MEP. The HFS group had the highest (σ_0); however, it did not show a statistical difference from MEP. Weibull distributions are shown in Table 3 and Figure 4.

Table 3: Tukey Test for the SBS (MPa) Means (Standard Deviations) and the Weibull Modulus (m), Characteristic Strength (σ_0), and, Respective CI (95%) for SBS of the Treatment Surfaces by Ceramic Material[a]

	Surface Treatment	Group Name	Shear Bond Strength (SBS) (MPa)	Weibull Modulus (*m*)	95% CI for *m*	Weibull Characteristic Strength (σ_o) (MPa)	95% CI for (σ_o)
Lithium Disilicate (e.max CAD)	HF+Silane	LD-HFS	18.67 ± 3.49 A	6.22 a	4.4-8.6	20.32 α	18.99-21.74
	MEP	LD-MEP	7.00±4.2 B	1.0 a	0.1-5.9	8.8 α	3.80-20.78
LithiumSilicate (Celtra Duo)	HF+Silane	LSC-HFS	16.81 ± 2.62 A	8.8 a	7.6-10.25	17.70 α	16.83-18.62
	MEP	LSC-MEP	14.12 ± 3.51 B	4.13 b	2.47-6.9	15.53 α	13.84-17.41
Lithium Silicate (Suprinity)	HF+Silane	LSS-HFS	16.33 ± 3.08 A	7.4 a	6.3-8.9	17.81 α	16.78-18.90
	MEP	LSS MEP	13.87 ± 2.52 B	6.6 a	5.3-8.1	15.30 β	14.32-16.35

Abbreviations: LSS, Vita Suprinity; LSC, Celtra duo; LD, Lithium disilicate.

[a] *Different uppercase letters indicate statistically significant difference for each material for the SBS. Different lowercase letters indicate statistically significant difference for each material for the Weibull modulus. Different Greek alphabet letters indicate statistically significant difference for each material for characteristic strength.*

Optical Profilometry

Kruskal–Wallis revealed that the "surface treatments" produced statistically different roughness according to each ceramic (p<0.0001). The HFS groups for all ceramics presented the highest mean roughness. Similarly, for all materials, MEP generated the lowest roughness values: LD-MEP (0.47±0.10 μm), LSS-MEP (0.16±0.01 μm), and LSC-MEP (0.39±0.08 μm). Means and standard deviation of roughness and the differences between the groups are expressed in Table 4.

The 3D surface topography images of the specimens subjected to all experimental surface treatment are illustrated in Figure 5.

Scanning Electronic Microscopy (SEM)

SEM-FEG images referring to LD, LSS, and LSC specimens without surface treatment (Figure 6) present a homogeneous surface with grooves due to the sanding process during preparation of blocks. For groups with HFS treatment (Figure 6), irregularities with numerous microporosities and grooves are seen as a result of the dissolution of the glassy phase, leading to an increase in surface roughness. For LSS and LSC, these features were more evident. On the other hand, MEP produced a smoother and more homogeneous surface without numerous microporosities, as observed by the HF groups (Figure 6).

Failure Analysis

The main failure type for the groups with the pretest failures during thermal aging was in the resin cement–ceramic interface (Score A). Complete adhesive failures at the cement–dentin interface (Score A) were more frequent for all groups of the LD ceramic (100%). For

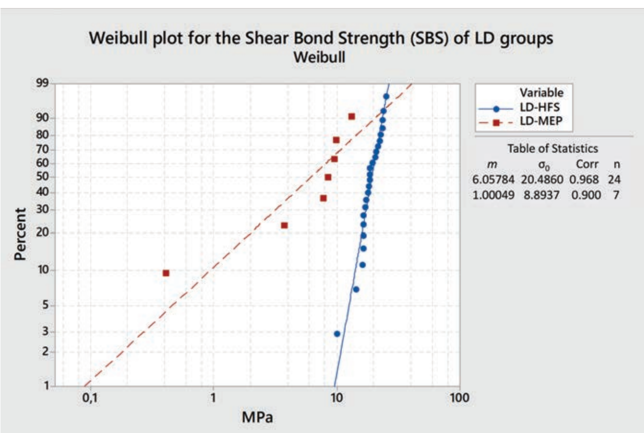

Figure 2. *Weibull plot for SBS of lithium disilicate (LD).*

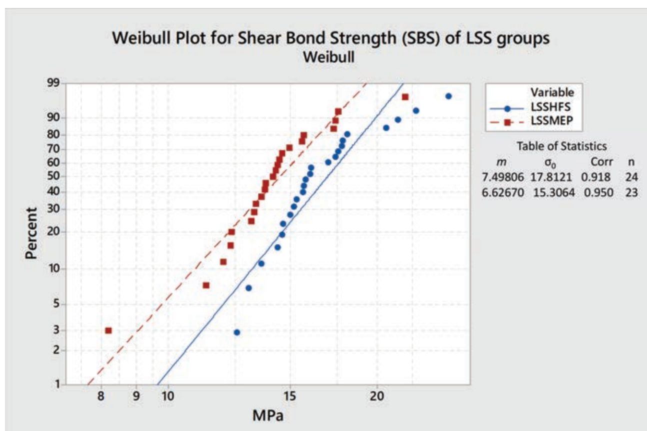

Figure 3. *Weibull plot for SBS of lithium silicate (LSS).*

Table 4: *Mean Roughness (R_a) Values (µm) with Standard Deviations for All Experimental Groups*[a]

Ceramic	Surface Treatment	Group Name	Mean Roughness (µm)
Lithium Disilicate (e.max CAD)	Control	C	0.29±0.05 B
	HF+Silane	LD-HFS	0.62±0.03 A
	MEP	LD-MEP	0.47± 0.10 B
Lithium Silicate (Celtra Duo)	Control	C	0.14± 0.02 B
	HF+Silane	LSC-HFS	1.33± 0.08 A
	MEP	LSC-MEP	0.39± 0.08 B
Lithium Silicate (Suprinity)	Control	C	0.24± 0.03 B
	HF+Silane	LSS-HFS	0.40± 0.02 A
	MEP	LSS MEP	0.16± 0.01 B

Abbreviations: LD: lithium disilicate; LSC: lithium silicate (Celtra Duo); LSS: lithium silicate (Suprinity).
[a] *Different uppercase letters show statistical differences among groups of each ceramic.*

the LSS and LSC groups, MEP also presented the greatest number of adhesive failures (Score A). After SBS, for the groups LSS-HFS (87.5%) and LSC-HFS (87.5%), the main failure type was Mixed 2 in the ceramic–resin cement interface + cohesive in ceramic (Score M2) (Table 2).

DISCUSSION

Many factors are involved in the clinical longevity of glass ceramic restorations,[31] such as the characteristics of the material used (composition, processing, and

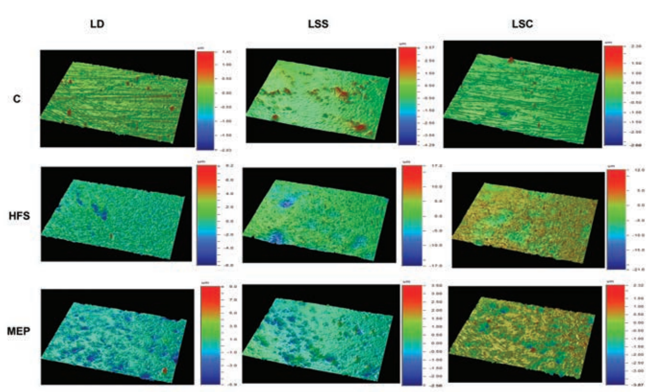

Figure 5. *3D Micrographs of surface topography (magnification 20×) of the specimens subjected to different surface treatment. LD: Lithium Disilicate; LSS: Vita Suprinity; LSC: Celtra Duo; C: Control (no treatment); HFS: Hydrofluoric acid + Silane; MEP: Monobond Etch & Prime/Ivoclar.*

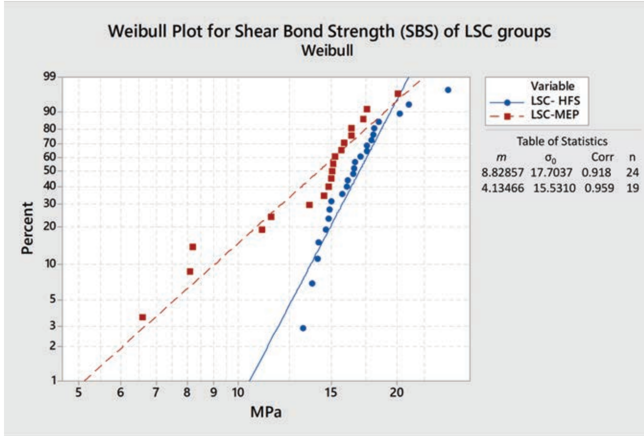

Figure 4. *Weibull plot for SBS of lithium silicate (LSC).*

thickness),[7,32] characteristics of the preparation and remaining substrate,[27,33] and the cementation protocol implemented,[31,34] which plays a key role in long-term adhesion. Thereby, the objective of this study was to evaluate the effect of different types of surface treatments on the adhesion of glass ceramics of *LD* and LS to resin cement. All the specimens in this study were subjected

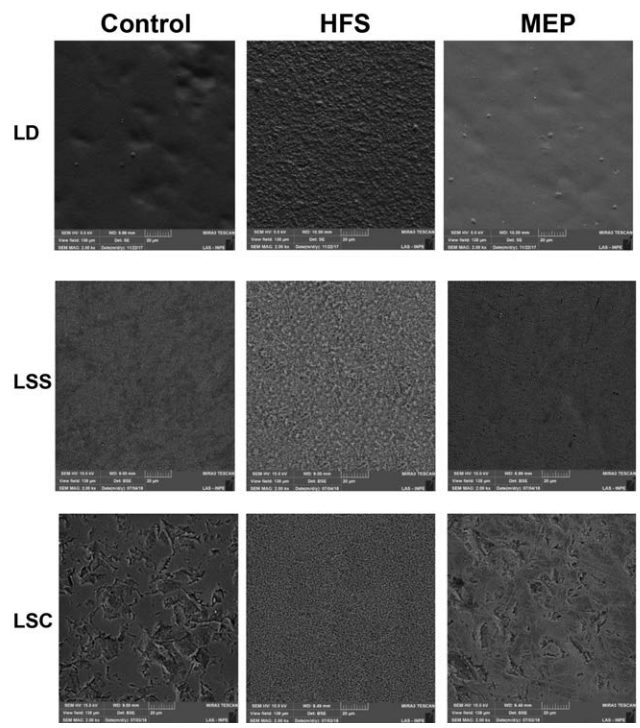

Figure 6. *Micrographs of SEM at 2500× magnification representing surface treatment groups. LD: Lithium Disilicate; LSS: Vita Suprinity; LSC: Celtra Duo; C: Control (no treatment); HFS: Hydrofluoric acid + Silane; MEP: Monobond Etch & Prime/Ivoclar.*

to thermocycling for 10,000 cycles, which simulates clinical conditions equivalent to 1 year of clinical use.[35]

The first hypothesis that the type of surface treatment would not influence the bond strength, regardless of the type of ceramic, was not accepted. In this study, different SBS values were found according to each material. Three glass ceramics were tested, and, despite the microstructural differences between these materials, the SBS results demonstrated that the HFS showed significantly higher adhesion values. Several studies have also shown that conditioning with HF followed by the application of silane was the best surface treatment for these glass ceramics.[5,7,13,14,22,36-39] HF etching causes micro-morphological changes and dissolves the glassy matrix creating micropores, where the resin cement can penetrate and provide micromechanical retention.[38,40] In addition, the application of a silane coupling agent to ceramic pretreated with HF provides a covalent chemical bond, which is one of the main factors of the efficient adhesion between glass ceramics and resin cements.[27] This treatment protocol offers the opportunity to improve micromechanical retention, and increase physical interactions and wettability with resin cement, which explains why it is the most suitable surface treatment.[41] According to some authors, this protocol, when balanced, prevents damage and weakening of the ceramic material, and dentists must follow the manufacturer's instructions for each ceramic, avoiding harmful effects of the material.[16,22]

Another surface treatment also investigated in this study was MEP. This treatment showed a significantly lower SBS for all ceramics investigated when compared to HF and higher SBS than SB and SC groups. Some authors report that MEP is less effective in glass ceramics than HF,[27,29,42] since this adhesive system contains ammonium polyfluoride—a milder acid with a lighter etching pattern that partially and homogeneously dissolves the glassy matrix[27]—generating a smoother surface alteration with less surface roughness and consequently lower adhesion values.[17,25,27,29,43-47] To compensate the pattern of conditioning, there has been suggested an active and prolonged application of MEP,[17,25] which leads to an increase in surface roughness, improving the interaction of resinous monomers (phosphoric acid methacrylate ester) with the ceramic surface and increasing the exposure of LD crystals with this acid.[25] Nevertheless, other authors state that the main MEP adhesion mechanism is chemical and, despite lower SBS in comparison to the HF etching, it seems to be a viable option, providing good adhesion values,[29] especially due to the simplification of procedures and in clinical situations of thin ceramic restorations.[28]

The second hypothesis that the surface treatment would influence the surface roughness regardless of the type of ceramic was not accepted. In this study, the HFS groups presented roughness higher than the MEP and control groups for all ceramics. Surface roughness is an important aspect that describes the effectiveness of pretreatment procedures on adhesion.[22,48] The literature shows that LS and LD materials have very similar microstructures. While LD presents small, needle-shaped crystals embedded in a glassy matrix, LS has slightly larger crystals with a more elongated, rounded, and rod-shaped appearance.[2,6] With regard to the HF group, the conditioning process selectively removes the glassy matrix, exposing the crystalline structure and generating a greater surface energy.[5,34] The SEM analysis revealed an irregular surface, with microporosity. Strasser and others[22] and Ramos and others[49] endorse that surfaces of glass ceramics etched with HF have strong and homogeneous corrosion patterns, resulting in a porous surface that favors adhesion. As a rule, roughness caused by MEP was slighter than the other treatments and statistically similar to the control groups. The SEM images of the MEP group specimens revealed less evident grooves; and the profilometry analysis proved that this treatment caused little increase in surface roughness (Figure 5). Strasser and others[22] confirm these small changes and suggest that this may also occur, because the primer present in the MEP itself caused the coating of surface irregularities and decreased R_a. Other authors also corroborate these findings.[27] Despite the lower performance compared to HF for LD ceramics, MEP also proved to be effective for LD, LSS, and LSC, presenting a high (m) and characteristic strength (σ_0) similar to HF, which reinforces the reliability of this adhesive interface, suggesting it as an option for surface treatment of glass ceramics, although further investigations should be carried out to complement these findings.

The failure analysis demonstrated different patterns of failures among treatments, with adhesive failures predominant in all surfaces of LD. For LSC and LSS, adhesive failures were more prevalent in group MEP. Whereas for HFS on the LSC and LSS ceramics, most of the failures were mixed (adhesive on the ceramic–cement interface and ceramic cohesive). Della and Northeast[50] claim that mixed failures can often occur due to the nonhomogeneous distribution of the SBS test, which generates stresses in the base materials. Contrastingly, the current study found this failure pattern more frequently only in groups with HFS. According to Baratto and others[31] and Mokhtarpour and others,[39] the presence of cohesive failures in ceramic demonstrate that the strength of the substrate and cement are equal to that of the adhesion area, which indicates a more effective surface treatment. In concern to MEP, as mentioned above, the adhesive failures can be related to the little mechanical retention that this material generates,[27] which was also confirmed in our results of profilometry and SEM.

In the present study, a large number of pretest failures were also observed during thermocycling for all the ceramics, but it most clearly affected the LD groups with MEP, which may justify the smaller mean values of SBS and greater standard deviation for these treatments. According to Cadernas and others,[17] MEP, when applied to LD with the recommended time, causes a greater number of adhesive failures. El-Damanhoury and others[27] indicated that the application of MEP on LD produces a low surface roughness, which may have contributed to this failure profile. Other studies did not find significant differences between HFS and MEP[43,51]; but no aging of the specimens was performed. Besides the roughness, thermocycling contributes to reducing adhesion to resin cement.[29,52] The temperature difference causes saturation of the cement, with a greater hydrolytic degradation of the adhesive interface,[53] which may also have contributed to the large number of pretest failures.

Further studies evaluating surface treatments of LS and LD ceramics, especially with MEP and HF etching at different application times, on the bond strength and flexural strength, must be carried out. Controlled and randomized clinical trials are also needed to assess the long-term behavior of these materials and to establish an ideal surface treatment for each ceramic material.

CONCLUSIONS

Based on the work presented, it seems reasonable to conclude the following:

- HF followed of silanization promote higher values of bonding strength for LD and LS ceramics.
- HFS promoted higher roughness than MEP for all the ceramics.

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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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