

RESEARCH AND EDUCATION

# Bonding effectiveness of tooth-colored materials to resin cement provided by self-etching silane primer after short- and long-term storage



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Ceramic restorations have become the preferred prosthetic treatment in many clinical situations because of good biocompatibility, adequate mechanical properties, and remarkable esthetics.<sup>1-4</sup> In addition, computer-aided design and computer-aided manufacturing (CAD-CAM) materials have made ceramic restorations more convenient and expeditious, as well as offering some advantages over traditional processes such as the elimination of human error.<sup>5</sup> CAD-CAM glass-ceramics are a suitable option when mechanical performance and high level esthetics are required.<sup>1,2</sup>

Glass-ceramics are composed of a silica-rich phase,<sup>6</sup> which can be reinforced with crystalline content to improve mechanical properties<sup>7</sup> or be infiltrated by a polymer structure seeking a decrease in crack growth rate, better shock absorption, and lower rigidity.<sup>8-13</sup> To improve bonding between these materials and composite resin

## ABSTRACT

**Statement of problem.** Glass-ceramic materials are typically treated with hydrofluoric acid (HF) and silane to improve their bond to composite resin; however, HF may be harmful to human tissues and the integrity of the material, and its application is a technique-sensitive procedure. A novel self-etching ceramic primer has been introduced with the claim that it can solve those problems. However, independent scientific evidence regarding its performance is scarce.

**Purpose.** The purpose of this in vitro study was to evaluate the effect of self-etching silane primer on glass-ceramic surface roughness and on long-term bonding between glass-ceramic and composite resin cement.

**Material and methods.** Plates of 3 materials (n=10), lithium disilicate glass-ceramic (LDC) (IPS e.max CAD), leucite-based glass-ceramic (LEU) (IPS Empress CAD), and resin-modified ceramic (PIC) (VITA ENAMIC), were treated in the following ways: no treatment (C), HF (5%) applied during the recommended time for each material (HF), and self-etching ceramic primer (Monobond Etch & Prime [MBEP]). Surface roughness (Sa) was analyzed with a laser 3D profiler. Ceramic sticks were subjected to (n=20) no treatment (C); treatment with hydrofluoric acid plus silane (HF+S); and treatment with self-etching ceramic primer (MBEP) bonded to prepolymerized composite resin sticks with composite resin cement (Variolink II) and stored for 24 hours and 1 year (n=10). The assemblies were submitted to microtensile bond strength testing ( $\mu$ TBS). Data were analyzed using ANOVA and the Tukey pairwise, post hoc test ( $\alpha=.05$ ). Failure pattern and surface and interface morphology were assessed using scanning electron microscopy.

**Results.** Only individual factors resulted in statistically significant differences for both variables (material:  $P<.001$ ; surface treatment:  $P=.020$ ), interaction ( $P=.570$ ). HF group ( $0.49 \pm 0.11 \mu\text{m}$ ) showed statistically higher roughness values ( $P\leq.05$ ) than control groups ( $0.44 \pm 0.97 \mu\text{m}$ ), while MBEP ( $0.48 \pm 0.11 \mu\text{m}$ ) was comparable with both. HF produced greater surface alterations than MBEP and C. PIC ( $0.60 \pm 0.051 \mu\text{m}$ ) exhibited significantly higher roughness values ( $P\leq.05$ ) than LDC ( $0.37 \pm 0.07 \mu\text{m}$ ) and LEU ( $0.45 \pm 0.04$ ). Regarding  $\mu$ TBS, the general mean of PIC ( $24.6 \pm 10.1 \text{ MPa}$ ) was higher ( $P\leq.05$ ) than LEUs ( $14.7 \pm 6.7 \text{ MPa}$ ) and LDCs ( $13.1 \pm 4.8 \text{ MPa}$ ), while treatment groups HF+S ( $17.9 \pm 10.0 \text{ MPa}$ ) and MBEP ( $20.5 \pm 9.7 \text{ MPa}$ ) produced higher  $\mu$ TBS values than control groups ( $14.2 \pm 5.5 \text{ MPa}$ ). Adhesive failure was associated with low  $\mu$ TBS values and aged specimens, while cohesive failure within the composite resin-cement layer and mixed failures were associated with higher  $\mu$ TBS values. Interface debonding was detected in C groups for LDC and LEU. PIC exhibited better interface stability.

**Conclusions.** MBEP produced smoother surfaces than HF. HF+S and MBEP significantly improved ceramic and composite resin cement bonding. (J Prosthet Dent 2019;121:713.e1-e8)

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## Clinical Implications

Simplified self-etching ceramic primer is a suitable alternative for glass-ceramic surface treatment as its bonding enhancing effectiveness is comparable with that of separate hydrofluoric acid and silane primer application while producing fewer alterations on the ceramic surfaces.

cements, they are typically treated with hydrofluoric acid (HF) and silane.<sup>14-16</sup> HF etching generates mechanical interlocking of the composite resin cement on the ceramic surface, and, as its performance depends on the composition of the substrate, different parameters have been recommended in terms of concentration and application time.<sup>17-19</sup> In addition, silane acts as a bridge between ceramic and composite resin-based materials, binding both (chemically) through 2 functional groups.<sup>14</sup> Silanization has been evaluated after simplifying and varying some application parameters to test silane behavior upon water degradation and as a bond enhancer.<sup>20,21</sup>

A novel ceramic primer has been recently introduced, claiming to etch and silanate glass-ceramics in a single step.<sup>22</sup> This self-etching ceramic primer is composed of a silane coupler along with a mild etchant (tetrabutylammonium dihydrogen trifluoride) that has been used in industrial processes.<sup>23</sup> According to the manufacturer, it is expected to produce a smoother etching than HF, in addition to providing a strong and long-lasting chemical bond.<sup>22</sup> Reports on the efficiency of this novel ceramic primer state that it may produce a bond strength similar to that of separate HF and silane applications,<sup>24,25</sup> in addition to producing less aggressive etching.<sup>24,26</sup> However, aspects of this simplified silane primer, such as long-term bonding effectiveness, effect of water degradation, bonded interface integrity submitted to aging, and the compatibility of this self-etching primer with recently developed resin-modified ceramics, have yet to be evaluated.

Thus, the purpose of this *in vitro* study was to evaluate the effect of a self-etching ceramic primer on the surface roughness of the treated material and the bond strength between a representative composite resin cement and 3 CAD-CAM glass-ceramics after short- and long-term storage. The null hypothesis tested was that no significant differences would be found on the surface roughness or bond strength produced by the surface treatments tested among the different materials stored in water for 24 hours or 1 year.

## MATERIAL AND METHODS

A lithium disilicate glass-ceramic (LDC) (IPS e.max CAD; Ivoclar Vivadent AG), a leucite-based glass-ceramic (LEU) (IPS Empress CAD; Ivoclar Vivadent AG), and a resin-modified ceramic (PIC) (VITA ENAMIC; VITA Zahnfabrik) were used. Seventy-two sticks (1.8×1.8×3.5 ±0.2 mm) and 30 plates (4.0×4.0×2.0 mm ±0.5 mm) were cut from CAD-CAM blocks of each material using a precision cutting machine and a diamond saw (Isomet 1000; Buehler) under constant irrigation. LDC specimens were sintered according to the instructions provided by the manufacturer (Austromat M; DEKEMA Dental-Keramiköfen GmbH). All specimens were polished with #1000 SiC paper and ultrasonically cleaned in distilled water for 5 minutes. Composition and instructions for the use of each material are summarized in Table 1.

Specimen sizes for both variables were calculated with a sample size calculation test (Minitab v17.2.1; Minitab Inc) with a confidence degree of 95%. The involved parameters were obtained from a pilot study. The minimum number of specimens obtained from this analysis was of 4 (roughness) and 9 ( $\mu$ TBS); therefore, both specimen numbers were standardized to n=10.

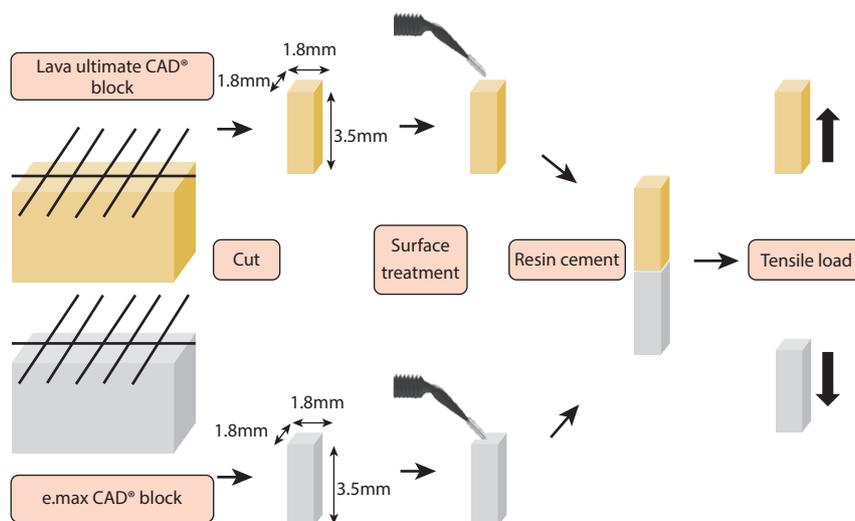
To evaluate surface roughness (Sa), ceramic plates from each material were distributed into 3 groups (n=10): control group or no treatment (C); HF (5%) applied during the recommended time for each material (LDC: 20 seconds, LEU and PIC: 60 seconds), washed with water for 60 seconds and ultrasonically cleaned for 5 minutes; and self-etching ceramic primer (Monobond Etch & Prime [MBEP]; Ivoclar Vivadent AG) in which the primer was applied on ceramic surfaces for 20 seconds actively, left to react for 40 seconds, and washed with water for 10 seconds (Table 1). The specimens were analyzed with a confocal optical 3D profiler (Lext OLS4000; Olympus Corp) at ×216 magnification, and a tridimensional roughness average (Sa) was calculated. Statistical analysis was performed with statistical software (Minitab v18; Minitab Inc) using 2-way ANOVA (material versus surface treatment) and the Tukey pairwise post hoc test ( $\alpha=.05$ ).

For the  $\mu$ TBS test, prepolymerized composite resin (Lava Ultimate CAD; 3M Oral Care) and the same glass-ceramic blocks were cut into sticks (1.8×1.8×3.5 ±0.2 mm). Ceramic sticks were treated by following the same parameters as for roughness evaluation: control (C), hydrofluoric acid and silane (HF+S), and self-etching ceramic primer (MBEP) (n=20). The only difference was that the sticks from group HF+S used for  $\mu$ TBS testing were also treated with silane (Monobond S; Ivoclar Vivadent AG) after HF (applied for 60 seconds and air-dried for 30 seconds). Prepolymerized composite

**Table 1.** Material descriptions

Material/Manufacturer	Type, Lot. No.*	Composition	Application Mode
IPS Empress CAD (LEU) (Ivoclar Vivadent AG)	Leucite-based glass-ceramic, U01096/A2	Leucite crystal: KAlSi <sub>2</sub> O <sub>6</sub> (35-45 %vol) Standard composition: SiO <sub>2</sub> (60-65 %wt), Al <sub>2</sub> O <sub>3</sub> (16-20 %wt), K <sub>2</sub> O (10-14 %wt), Na <sub>2</sub> O (3.5-6.5 %wt), other oxides (0.5-7 %wt), pigments (0.2-1 %wt)	
IPS e.max CAD (LDC) (Ivoclar Vivadent AG)	Lithium disilicate-reinforced glass-ceramic, T25503/A2	SiO <sub>2</sub> (57-80 %wt), Li <sub>2</sub> O (11-19 %wt), K <sub>2</sub> O (0-13 %wt), P <sub>2</sub> O <sub>5</sub> (0-11 %wt), ZrO <sub>2</sub> (0-8 %wt), ZnO (0-8 %wt), Al <sub>2</sub> O <sub>3</sub> (0-5 %wt) MgO (0-5 %wt), coloring oxides (0-8 %wt)	
VITA ENAMIC (PIC) (VITA Zahnfabrik)	Resin-modified ceramic, 43230/A2	Fine-structure feldspar ceramic network (86 %wt/75 %vol): SiO <sub>2</sub> (58-63 %wt), Al <sub>2</sub> O <sub>3</sub> (20-23 %wt), Na <sub>2</sub> O (6-11 %wt), K <sub>2</sub> O (4-6 %wt), B <sub>2</sub> O <sub>3</sub> (0.5-2 %wt), ZrO <sub>2</sub> (<1 %wt), CaO (<1 %wt), TiO <sub>2</sub> (<1 %wt) Methacrylate-polymer network (14 %wt/25 %vol): urethane dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA)	
Power C Etching 5% (HF) (BM4/Materiais e Instrumentais Ltda.)	5% hydrofluoric acid	Hydrofluoric acid, thickener, surfactant, water	Apply on ceramic surface for indicated time and thoroughly wash with water
Monobond Etch & Prime (MBEP) (Ivoclar Vivadent AG)		Butanol, tetrabutylammonium dihydrogen trifluoride, methacrylated phosphoric acid ester, bis(triethoxysilyl) ethane, silane methacrylate, colourant, ethanol, water	Actively apply on ceramic surface for 20 s, let it react for 40 s, and wash with water for 10 s
Monobond S (S) (Ivoclar Vivadent AG)		Ethanol, water, silane methacrylate	Apply to conditioned ceramic surface with microbrush, let it react for 60 s, and blow using strong air stream

\*Data taken from each material safety data sheet or technical data information provided by manufacturers.



**Figure 1.** Schematic representation of microtensile bond strength test arrangement.

resin sticks were airborne-particle abraded (50- $\mu$ m aluminum oxide, 200 kPa pressure, 10 seconds, 10-mm distance), ultrasonically cleaned in alcohol for 5 minutes, dried with oil-free air, and coated with an adhesive system (Scotchbond Universal; 3M Oral Care). Then, the composite resin and ceramic sticks were cemented (Variolink II; Ivoclar Vivadent AG) with the aid of a custom jig and magnification ( $\times 15$ ). Each face was light polymerized for 20 seconds (Bluephase; Ivoclar Vivadent AG; 1200 mW/cm<sup>2</sup>), and groups were divided into 2 subgroups for storage purposes (37°C distilled water storage): 24 hours and 1 year (Fig. 1). Two more specimens from each group/material were fabricated to

analyze bonded interfaces. After aging, each specimen was submitted to a tensile load (universal testing machine [EZ Test; Shimadzu Corp], 50-N load cell, cross-head speed: 1.0 mm/min).  $\mu$ TBS data were expressed in MPa and statistically analyzed by applying the 3-way ANOVA (surface treatment versus storage time versus material) and the Tukey post hoc tests. Pretesting bond failures were not considered in the statistical analysis. In addition, the Pearson correlation test was performed between roughness and bond strength ( $\alpha=.05$ ).

The ceramic plates used to evaluate roughness, the debonded sticks from  $\mu$ TBS, and the bonded sticks for interface analysis were mounted on aluminum stubs and

**Table 2.** Roughness mean values (Sa) ±standard deviations (μm)

Material	Surface Treatment			Tukey-material ( $P \leq .05$ )
	C	HF	MBEP	
LDC	0.34 ±0.07	0.39 ±0.05	0.37 ±0.06	0.37 ±0.07 C
LEU	0.44 ±0.02	0.45 ±0.05	0.46 ±0.04	0.45 ±0.04 B
PIC	0.54 ±0.05	0.63 ±0.02	0.61 ±0.03	0.60 ±0.05 A
Tukey-surface treatment ( $P \leq .05$ )	0.441 ±0.97 B	0.49 ±0.11 A	0.48 ±0.11 AB	

C, control group; HF, hydrofluoric acid; LDC, lithium disilicate ceramic (IPS e.max CAD); LEU, leucite-based ceramic (IPS Empress CAD); MBEP, Monobond Etch & Prime; PIC, resin-modified ceramic (VITA ENAMIC). Only individual factors resulted in statistically significant differences (material, surface treatment), and no significant interaction between factors was recorded. General means from each level displayed in separate column (material) or row (surface treatment) for statistical comparisons. Different uppercase letters represent statistical differences within levels of each individual factor (Tukey test,  $P \leq .05$ ).

sputter coated with gold/palladium alloy powder to be examined with scanning electron microscopy (JSM 5600 LV; JEOL Ltd), operating at 15 kV and a working distance of 20 mm. Representative images were captured at different magnifications. Fractured surfaces were classified as adhesive failure between the ceramic and composite resin cement; cohesive-ceramic failure as failure within the ceramic material; cohesive-composite resin cement failure as fracture within the composite resin cement; and mixed failure as a mixture of different fractures in the same area.

## RESULTS

For the statistical analysis (Sa) of surface roughness, normality (Anderson-Darling test:  $P=.163$ ) and homoscedasticity (Bartlett test:  $P=.328$ ) were demonstrated. ANOVA revealed that both factors significantly influenced roughness values (material:  $P<.001$ ; surface treatment:  $P=.020$ ), but no interaction was reported between factors ( $P=.570$ ). Roughness values are summarized in Table 2. PIC obtained the highest roughness mean values and surface alterations, followed by LEU, while LDC showed the lowest. The control groups had the lowest surface roughness, and the groups treated with HF had the greatest surface roughness. LDC had fewer surface alterations (Fig. 2). HF produced multiple irregularities on PIC specimens, exposing the polymer structure, but MBEP groups showed a smoother appearance (Fig. 2).

Microtensile bond strength data were transformed using the Box-Cox procedure ( $\lambda:0.033$ ) to achieve positive normality (Anderson-Darling test:  $P=.070$ ) and homoscedasticity (Bartlett test:  $P=.070$ ). All factors significantly influenced  $\mu$ TBS values (material:  $P<.001$ ; surface treatment:  $P=.001$ ; storage time:  $P=.030$ ), but no interaction was found between factors ( $P>.05$ ).  $\mu$ TBS data are displayed in Table 3. Only the control groups (LDC and LEU) showed pretesting failures (100%). Within the factor “material,” PIC obtained the highest

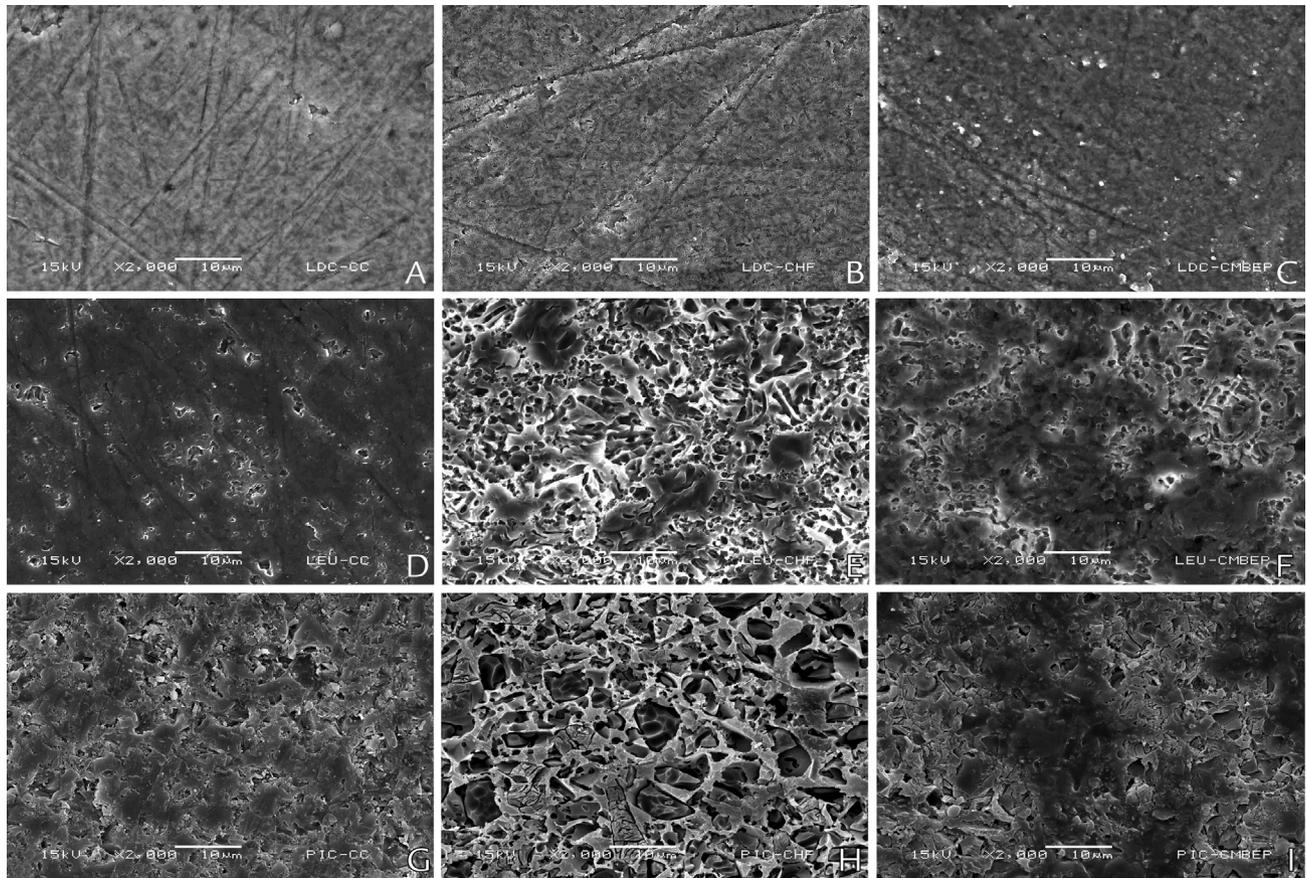
$\mu$ TBS values, while in “surface treatment” groups, HF+S and MBEP showed higher  $\mu$ TBS values than the control group. The aged groups showed lower  $\mu$ TBS values compared with the 24-hour groups. No significant correlation was detected between the variables (roughness and  $\mu$ TBS) or factors, as all Pearson correlation coefficients remained below 0.333, and all  $P$  values were higher than .05.

Failure patterns are summarized in Table 4 and represented in Figure 3. In general, a high prevalence of adhesive failures was found in the 1-year aged specimens. Cohesive failures in composite resin cement and mixed failures were associated with higher  $\mu$ TBS values (Tables 3 and 4). Interface analysis showed complete debonding between the ceramic material and the composite resin cement in the control groups. PIC reported better interlocking with the composite resin cement (Fig. 4).

## DISCUSSION

The present study found that the surface treatment of the ceramic, the composition of such ceramic material, and the storage conditions affected the surface roughness of the ceramic surface and the microtensile bond strength between the ceramic and composite resin cement. Therefore, the null hypothesis was rejected.

The surface of glass-ceramics should be altered to produce microroughness, stimulating the interlocking of an adhesive cementation material onto its structure, increasing the surface energy of the ceramic, and improving the bonding performance between both materials.<sup>14,15</sup> In this study, self-etching silane primer (MBEP) produced a milder surface etching on the ceramic surface when compared with HF (Fig. 2). This may be explained by the presence of tetrabutylammonium dihydrogen trifluoride on MBEP, acting as the etchant agent.<sup>22</sup> Ammonium polyfluoride salts have been used in industrial processes to etch silica-based materials and have produced smoother etching patterns than HF.<sup>23</sup> Thus, a less aggressive glassy phase dissolution was expected on the specimens treated with MBEP as confirmed in the present work and in previous studies.<sup>24,26</sup> Differences in the surface morphology were greater on LEU and PIC than on LDC (Fig. 2). This may be because of the greater HF application time on LEU and PIC (60 seconds) than on LDC (20 seconds), as the manufacturer’s instructions were followed. Increasing etching time has been related to greater glassy phase removal on glass-ceramic materials, also depending on the composition of the ceramic.<sup>17,18</sup> LDC may have had less surface damage (Fig. 2) because it has higher crystalline content.<sup>7</sup> Similarly, because of the mixture of ceramic and polymer, PIC showed a more irregular surface,<sup>9,10,12</sup> which means less time was



**Figure 2.** Representative scanning electron microscope images regarding surface morphology produced by each treatment (original magnification  $\times 2000$ ). A, LDC/C; B, LDC/HF; C, LDC/MBEP; D, LEU/C; E, LEU/HF; F, LEU/MBEP; G, PIC/C; H, PIC/HF; I, PIC/MBEP. C, control group; HF, hydrofluoric acid; LDC, lithium disilicate ceramic (IPS e.max CAD); LEU, leucite-based ceramic (IPS Empress CAD); MBEP, Monobond Etch & Prime; PIC, resin-modified ceramic (VITA ENAMIC).

**Table 3.** Microtensile bond strength values (MPa)  $\pm$  standard deviations and pretesting failure rates (ptf)

Material	Surface Treatment and Storage Time						Tukey-material
	C		HF+S		MBEP		
	24 h	1 y	24 h	1 y	24 h	1 y	
LDC	-	-	13.5 $\pm$ 1.8	10.0 $\pm$ 3.1	15.5 $\pm$ 6.6	13.4 $\pm$ 5.3	LDC: 13.1 $\pm$ 4.8 B
	ptf: 10/10	ptf: 10/10	ptf: 0/10	ptf: 0/10	ptf: 0/10	ptf: 0/10	
LEU	-	-	13.0 $\pm$ 3.9	12.1 $\pm$ 2.2	20.4 $\pm$ 11.0	13.4 $\pm$ 1.4	LEU: 14.7 $\pm$ 6.7 B
	ptf: 10/10	ptf: 10/10	ptf: 0/10	ptf: 0/10	ptf: 0/10	ptf: 0/10	
PIC	16.6 $\pm$ 5.2	11.7 $\pm$ 5.0	29.6 $\pm$ 10.0	29.1 $\pm$ 8.5	32.3 $\pm$ 5.2	28.1 $\pm$ 6.2	PIC: 24.6 $\pm$ 10.1 A
	ptf: 0/10	ptf: 0/10	ptf: 0/10	ptf: 0/10	ptf: 0/10	ptf: 0/10	
Tukey "surface treatment"	C: 14.2 $\pm$ 5.5 B		HF+S: 17.9 $\pm$ 10.0 A		MBEP: 20.5 $\pm$ 9.7 A		
Tukey "storage time"			24 h: 15.6 $\pm$ 7.1 A	1 y: 12.3 $\pm$ 3.5 B			

C, control group; HF+S, hydrofluoric acid+silane; LDC, lithium disilicate ceramic (IPS e.max CAD); LEU, leucite-based ceramic (IPS Empress CAD); MBEP, Monobond Etch & Prime; PIC, resin-modified ceramic (VITA ENAMIC); ptf, pretesting failures. Only individual factors resulted in statistically significant differences (material, surface treatment, storage time), and no significant interaction between factors was recorded. General means from each level along with Tukey test results (uppercase letters) are displayed next to each name in separate rows (surface treatment/storage time) or columns (material) for statistical comparisons. Different letters represent statistical differences within levels of each individual factor (Tukey test,  $P \leq .05$ ).

needed for the etchant to act and expose the polymer network (Fig. 2).

The bonding effectiveness promoted by different surface treatments was evaluated using microtensile bond strength testing. Individual sticks were built up to avoid possible additional tensile stress induced by traditional

cementing and cutting procedure. These results show that MBEP and HF+S perform similarly, as reported previously.<sup>24,25</sup> Even though HF produced higher surface roughness than MBEP, both treatments promoted similar bonding potential. Therefore, high roughness is not always related to high bond strength values.

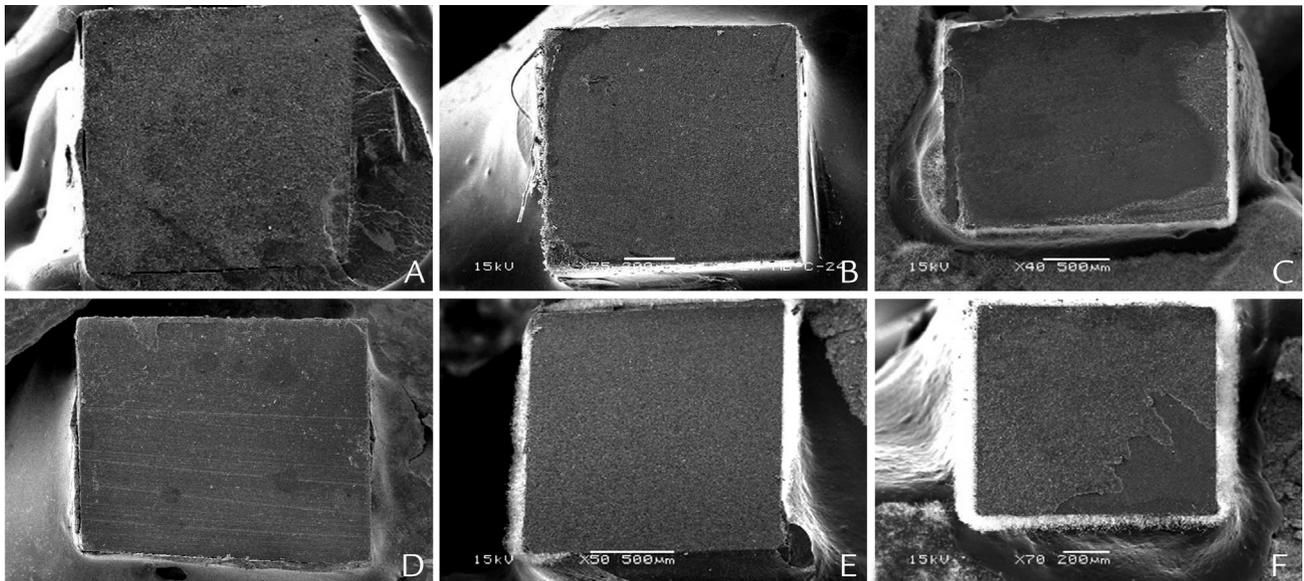
**Table 4.** Failure mode prevalence among experimental groups

Material	Groups/Failure Type	Adhesive	Cohesive-Ceramic	Cohesive-Composite Resin Cement	Mixed		
LDC	C/24 h	Number of specimens	0	0	0	0	
		Percentage (%)	0	0	0	0	
	HF+S/24 h	Number of specimens	4	0	4	2	
		Percentage (%)	40	0	40	20	
	MBEP/24 h	Number of specimens	1	0	4	5	
		Percentage (%)	10	0	40	50	
	C/1 y	Number of specimens	0	0	0	0	
		Percentage (%)	0	0	0	0	
	HF+S/1 y	Number of specimens	6	0	2	2	
		Percentage (%)	60	0	20	20	
	MBEP/1 y	Number of specimens	1	0	2	7	
		Percentage (%)	10	0	20	70	
	LEU	C/24 h	Number of specimens	0	0	0	0
			Percentage (%)	0	0	0	0
HF+S/24 h		Number of specimens	5	0	5	0	
		Percentage (%)	50	0	50	0	
MBEP/24 h		Number of specimens	1	0	5	4	
		Percentage (%)	10	0	50	40	
C/1 y		Number of specimens	0	0	0	0	
		Percentage (%)	0	0	0	0	
HF+S/1 y		Number of specimens	7	0	1	2	
		Percentage (%)	70	0	10	20	
MBEP/1 y		Number of specimens	4	0	4	1	
		Percentage (%)	40	0	40	10	
PIC		C/24 h	Number of specimens	10	0	0	0
			Percentage (%)	100	0	0	0
	HF+S/24 h	Number of specimens	2	0	6	2	
		Percentage (%)	20	0	60	20	
	MBEP/24 h	Number of specimens	1	0	5	4	
		Percentage (%)	10	0	50	40	
	C/1 y	Number of specimens	10	0	0	0	
		Percentage (%)	100	0	0	0	
	HF+S/1 y	Number of specimens	4	0	5	1	
		Percentage (%)	40	0	50	10	
	MBEP/1 y	Number of specimens	2	0	1	7	
		Percentage (%)	20	0	10	70	

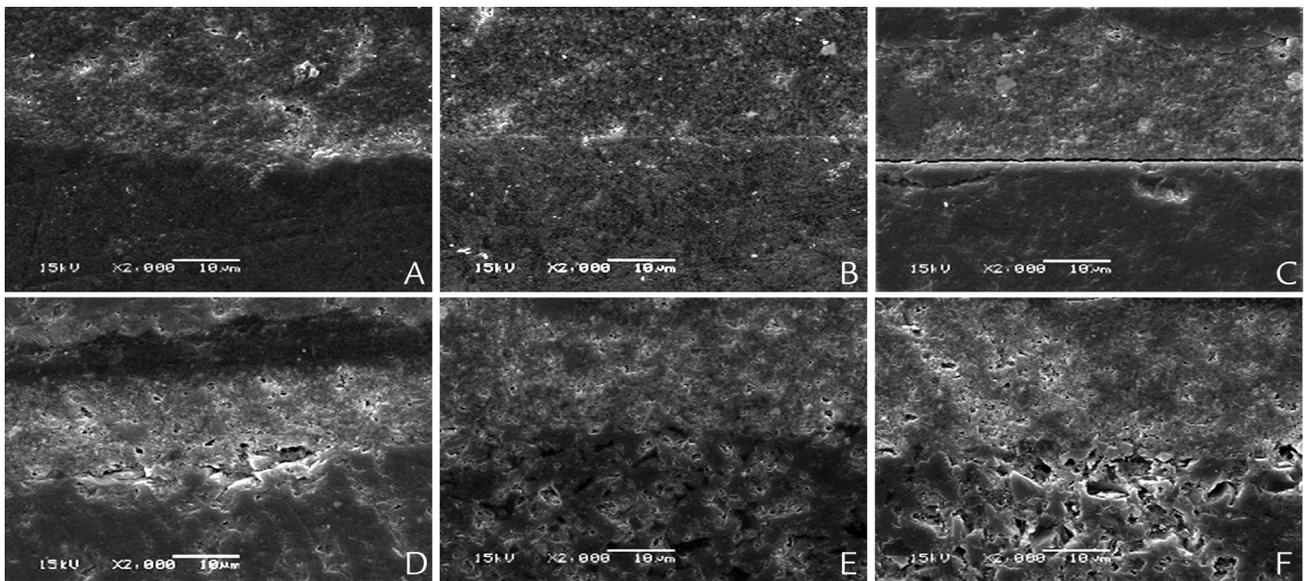
C, control group; HF+S, hydrofluoric acid+silane; LDC, lithium disilicate ceramic (IPS e.max CAD); LEU, leucite-based ceramic (IPS Empress CAD); MBEP, Monobond Etch & Prime; PIC, resin-modified ceramic (VITA ENAMIC).

As MBEP was as effective as HF+S, combining a mild surface etchant with a stable silane primer in 1 single solution may be efficient. The manufacturer recommends applying MBEP on the ceramic surface for 20 seconds, letting it react for 40 seconds, and then washing it with water. The authors are unaware of other silane primers for which washing with water is recommended by the manufacturer, even though silane chemical bonding has been defined as a water-resistant union.<sup>14,20</sup> The rationale of performing this step may be to eliminate the acid etchant from the ceramic surface and to leave only the silane agent. Apparently, the silane contained in MBEP could form a water-resistant chemical union between the glassy substrate and the composite resin cement. This can be related to the high

prevalence of mixed and composite resin-cement cohesive failures exhibited by MBEP. HF+S probably suffered a greater effect of water degradation on the bonded interface as it showed a greater number of adhesive failures (Table 4, Fig. 3). A possible explanation may be the incomplete removal of solvent and reaction byproducts after the application of conventional silane. The time recommended by the manufacturer for air blowing has been reported to be insufficient to eliminate those products from the silane layer.<sup>16,20,21</sup> The water-cleaning step (MBEP) may have removed the solvent and byproducts from the ceramic surfaces better than the air-blowing step (HF+S); by chemical affinity, water would better collect and eliminate such substances.



**Figure 3.** Scanning electron microscope images regarding failure pattern from some groups (LEU/MBEP 24 hours, PIC/MBEP 24 hours, LDC/HF+S 1 year, LEU/HF+S 1-year, PIC/C 1 year, PIC/MBEP 1 year), exhibited on ceramic side. A, LEU/MBEP, 24 hours: cohesive failure in resin cement (original magnification  $\times 75$ ); B, PIC/MBEP, 24 hours: cohesive failure in resin cement (original magnification  $\times 75$ ); C, LDC/HF+S, 1 year: adhesive failure between ceramic and cement (original magnification  $\times 75$ ); D, LEU/HF+S, 1-year: adhesive failure between ceramic and cement (original magnification  $\times 50$ ); E, PIC/C, 1 year: adhesive failure (original magnification  $\times 50$ ); F, PIC/MBEP, 1-year: mixed failure (original magnification  $\times 50$ ). C, control group; HF+S, hydrofluoric acid+silane; LDC, lithium disilicate ceramic (IPS e.max CAD); LEU, leucite-based ceramic (IPS Empress CAD); MBEP, Monobond Etch & Prime; PIC, resin-modified ceramic (VITA ENAMIC).



**Figure 4.** Representative SEM images of bonded interfaces from some groups (original magnification  $\times 2000$ ). A, LDC/HF+S, 24 hours; B, LDC/MBEP, 24 hours; C, LEU/C, 1 year; D, LEU/MBEP, 1 year; E, PIC/C, 24 hours; F, PIC/C, 1 year. C, control group; HF+S, hydrofluoric acid+silane; LDC, lithium disilicate ceramic (IPS e.max CAD); LEU, leucite-based ceramic (IPS Empress CAD); MBEP, Monobond Etch & Prime; PIC, resin-modified ceramic (VITA ENAMIC).

Some partial debonded areas were noted on some ceramic/cement interfaces (LDC and LEU), but such defects seemed to originate within the composite resin cement layer (Fig. 4), possibly due to the dislodgment of filler particles because of water degradation and poor

ceramic-cement interaction. Conversely, PIC showed a good interface integrity (Fig. 4) in addition to obtaining the highest  $\mu$ TBS values and showing no pretesting failures (Table 3), which may indicate this material bonds reliably with composite resin cement.

Limitations of this in vitro study include the use of a prepolymerized composite resin as a substrate for bonding the ceramic material (instead of dentin) and the separate fabrication of each stick, which resulted in a laborious and difficult process. Furthermore, aging in distilled water may not reflect the actual degradation dynamic of the evaluated interface in the oral environment. Despite these limitations, self-etching ceramic primer can be recommended for bonding glass-ceramic to composite resin cement. Simplified ceramic primers are promising as they appear to be effective and more time saving than standard procedures. However, further research regarding other aspects of this novel self-etching ceramic primer should be addressed, for example, chemical and mechanical analysis of bonding stability, variations on application protocols, interaction with different types of composite resin cements, and clinical performance.

## CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The self-etching silane primer provided a similar ceramic/cement bond strength to HF etching and silane application.
2. Both treatments were affected by water storage.
3. The self-etching ceramic primer produced lower surface roughness and fewer morphological alterations than the standard HF etching.
4. The novel composite resin-modified ceramic (PIC) exhibited better bonding with composite resin cement and higher inherent surface roughness than lithium disilicate-reinforced ceramic or leucite-based ceramic.

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