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Surface/Interface Morphology and Bond Strength to Glass Ceramic Etched for Different Periods

LZ Naves • CJ Soares • RR Moraes
LS Gonçalves • MAC Sinhoreti • L Correr-Sobrinho

Clinical Relevance

Increased etching periods may impair the bond strength to ceramics, while the use of an unfilled resin after silane may improve bond strength. The application of a bonding resin may also provide better infiltration to the irregularities created on etched surfaces, irrespective of the conditioning time.

Lucas Z Naves, DDS, MS, PhD student, Department of Restorative Dentistry, Dental Materials Division, Piracicaba Dental School, University of Campinas-UNICAMP, Piracicaba, SP, Brazil

Carlos J Soares, DDS, MS, PhD, professor, Biomechanic Research Group, School of Dentistry, Federal University of Uberlândia, MG Brazil

Rafael R Moraes, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Federal University of Pelotas, RS, Brazil

Luciano S Gonçalves, DDS, MS, PhD student, Department of Restorative Dentistry, Dental Materials Division, Piracicaba Dental School, University of Campinas-UNICAMP, Piracicaba, SP, Brazil

Mário Alexandre C Sinhoreti, DDS, MS, PhD, professor, Department of Restorative Dentistry, Dental Materials Division, Piracicaba Dental School, University of Campinas-UNICAMP, Piracicaba, SP, Brazil

*Lourenço Correr-Sobrinho, DDS, MS, PhD, professor, Department of Restorative Dentistry, Dental Materials Division, Piracicaba Dental School, University of Campinas-UNICAMP, Piracicaba, SP, Brazil

*Reprint request: Av Limeira 901, Piracicaba, SP 13414-903, Brazil; e-mail: sobrinho@fop.unicamp.br

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SUMMARY

This study evaluated the influence of etching periods on the surface/interface morphology and bond strength to glass ceramic with or without application of an unfilled resin after silane. Ceramic discs were divided into 12 groups, defined by etching time with 10% hydrofluoric acid: G1/G7 – etching for 10 seconds, G2/G8 – 20 seconds; G3/G9 – 40 seconds; G4/G10 – 60 seconds; G5/G11 – 120 seconds and G6/G12–60 + 60 seconds. All the groups were silanated after etching and G7 – G12 received a layer of unfilled resin after silane. Microshear testing using resin cement was performed, with 12 resin cylinders tested per group. The data was submitted to two-way ANOVA and the Student-Newman-Keuls' test (*p*<0.05). Evaluation of the etching pattern and bonding interfaces was conducted by SEM. The bond strength means (MPa) were: 19.4 ± 3.5, 22.3 ± 5.1, 22.2 ± 3.2, 17.8 ± 2.1, 15.3 ± 3.0 and 14.3 ± 1.8 for G1–G6 and 17.4 ± 4.8, 21.3 ± 2.1, 21.1 ± 2.3, 24.7 ± 5.8, 20.4 ± 2.2 and 18.5 ± 4.6 for G7–G12. Poor etching was detected after 10 seconds of conditioning; whereas deep channels were extensively

observed on surfaces etched for 120 and 60 + 60 seconds. Unfilled voids underlying the ceramic-cement interface were detected when only silane was applied. Full completion of the irregularities on G11 was detected using unfilled resin. When only silane was applied, the 60-second group and those etched for longer periods showed lower bond strengths. When both silane and unfilled resin were applied, all etching periods generally showed similar values. In conclusion, the etching period influenced the surface/interface topography and bond strength to ceramic. The application of unfilled resin was able to infiltrate all unfilled voids beneath the ceramic-cement interface, except on re-etched surfaces.

INTRODUCTION

Acid-sensitive ceramics present a high content of vitreous phase. Among them, IPS Empress (Ivoclar Vivadent, Schaan, Liechtenstein) figures prominently as a hot-pressed leucite-reinforced material, with properties well reported in the literature.[1-4] However, there are few studies evaluating the recently introduced Empress Esthetic ceramic[5-7] that present finer leucite grains distributed in a more homogeneous manner than their predecessor.

The surface modification of glass ceramics by etching results in increased contact surface area, improving the interaction between the ceramic and the luting agent.[8-9] The number and size of the irregularities created have been associated with etching time,[10-12] acid formulation and dilution of the acid solution.[9,13] Empress Esthetic ceramic (Ivoclar Vivadent) is recommended to be conditioned for 60 seconds.[14] However, little is known about the effects of decreased or increased etching periods on the surface morphology or bonding ability of this ceramic. It is known that, although longer conditioning periods may increase the ceramic bond strengths,[11] the increase is dependent on the crystalline content of the material.[10] Longer etching periods may also impair complete infiltration of the irregularities by the resin cement; this scenario could be worsened if the clinician re-etches the ceramic, seeking higher retention and increasing the in-depth dissolution.

To take full advantage of the increased energy on etched surfaces, the cement needs sufficient wettability to completely infiltrate the irregularities.[15-17] Most manufacturers recommend the use of resin cements directly on the internal silanated ceramic surface. It remains questionable, however, whether the silane coupling agent and the cement are efficient in properly wetting the surface and filling up all irregularities when increased etching periods are used. This is especially of concern considering that the internal surfaces of indirect restorations are commonly irregular. Although some clinicians apply a thin layer of unfilled resin after the silane, the current literature still lacks information about this approach for luting purposes. It is possible that the use of this low-viscosity material would improve adaptation of substrates along the ceramic-polymer interface, as well as the bonding strengths, but these effects still warrant investigation.

The current study evaluated the bond strength of a dual-cured resin cement to a hot-pressed, leucite-reinforced glass ceramic after different etching periods, using or not using a bonding resin after silane application. This study also characterized the morphology of the etched surfaces and the interfaces created between the substrates involved. The hypothesis tested was that longer etching periods would create more surface irregularities and increase bond strengths to the ceramic.

METHODS AND MATERIALS

Ceramic Specimens

Leucite-reinforced glass ceramic discs of Empress Esthetic, shade ETC2 (Ivoclar Vivadent) 6 mm in diameter and 1-mm thick, were made in accordance with the manufacturer's instructions, as previously described.[7] The ceramic samples were embedded in epoxy resin and wet-polished with 400-, 600- and 1200-SiC abrasive papers in order to create a smooth, flat surface.

Etching Periods and Surface Treatment

The ceramic specimens (72 in total) were divided into 12 groups defined by the etching period, using 10% hydrofluoric (HF) acid gel. Table 1 presents a description of the tested groups. For the re-etched groups (60 + 60 seconds), the specimens were rinsed with distilled water and dried with air before the second etching procedure. For example, the re-etching procedure was included to simulate a clinical situation where the clinician reapplies the conditioning agent for increased retention. After etching, all the samples were rinsed with air/water spray for 20 seconds and dried with air

Table 1: *Description of the Tested Groups*

Group	Etching Time	Surface Treatment
G1	10 seconds	silane
G2	20 seconds	silane
G3	40 seconds	silane
G4	60 seconds	silane
G5	120 seconds	silane
G6	60 + 60 seconds	silane
G7	10 seconds	silane + unfilled resin
G8	20 seconds	silane + unfilled resin
G9	40 seconds	silane + unfilled resin
G10	60 seconds	silane + unfilled resin
G11	120 seconds	silane + unfilled resin
G12	60 + 60 seconds	silane + unfilled resin

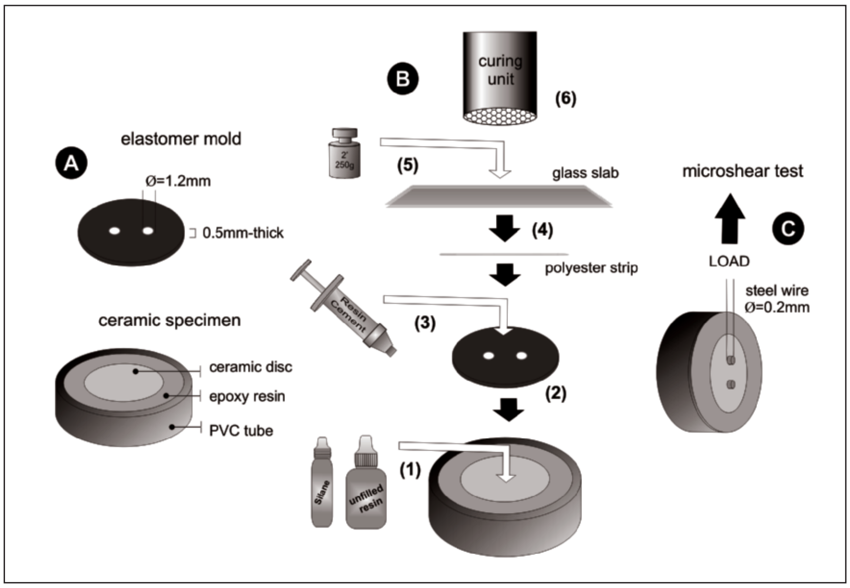

Figure 1: *Experimental setup of the study. After etching, (1) silane was applied to the ceramic surface, and for half of the specimens, the unfilled resin was applied after silane; (2) an elastomer mold with cylinder-shaped orifices was positioned onto the surface and photo-activation of the unfilled resin was performed; (3) orifices filled with resin cement; (4) polyester strip and glass slab placed over the filled mold; (5) cementation load applied for two minutes; (6) photo-activation of the resin cement; (7) samples were positioned on the testing machine and submitted to microshear testing.*

for 10 seconds. A silane coupling agent was applied on the surfaces for 10-15 seconds and air-dried for 60 seconds to allow effective removal of the solvent. In half of the specimens in each group, a thin layer of photo-activated unfilled resin (3M ESPE, Seefeld, Germany) was applied. The primer of the Scotchbond system was not used.

Bond Strength and Failure Analysis

In order to obtain specimens for the microshear test, the setup shown in Figure 1 was carried out.[6] Elastomer molds (0.5-mm thick), each with two cylinder-shaped orifices (1.2 mm in diameter), were placed onto the ceramic surfaces, enabling delimitation of the bonding area. For the groups where the unfilled resin was applied, photo-activation was performed for 20 seconds using a halogen curing unit. Photo-activation was conducted before insertion of the resin cement. Equal volumes of base and catalyst pastes of Variolink II dual-cured resin cement (Ivoclar Vivadent), shade A3, were mixed for 15 seconds; the cement was used to fill the orifices and a polyester strip was placed over the filled mold. A 250 g cementation load was applied for two minutes.[6] The cement was photo-activated for 40 seconds and the samples were stored in distilled water at 37°C for 24 hours. Two cylinders were built-up on each ceramic disc, with 12 cylinders tested in total for each group. Each resin cylinder was considered an independent experimental unit for the statistical analysis, because separate bonding procedures were performed to obtain the two cylinders for each disc.

Before the bond strength test, all resin cylinders were checked under magnification (x40) for bonding defects. Specimens presenting any irregularity at the boundaries of the cylinder were eliminated. A thin steel wire (0.2 mm in diameter) was looped around each cylinder and aligned with the bonding interface. The microshear test was conducted on a mechanical testing machine at a crosshead speed of 0.5 mm minute^{-1} until failure. The bond strength values were calculated in MPa. Bond strength data were submitted to two-way ANOVA and Student-Newman-Keuls' test (p<0.05). The fractured specimens were examined under optical microscopy at a 40× magnification. The failure mode was classified as adhesive, cohesive within ceramic or mixed, involving bonding agent, cement and ceramic.

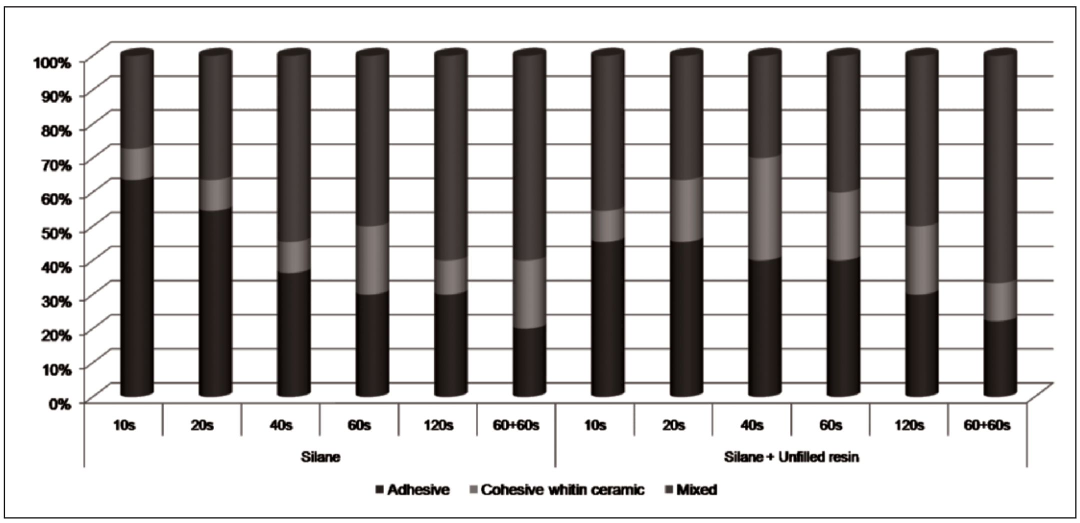

Figure 2: *Distribution of failure modes (%) among groups. A decrease in adhesive failures and an increase in mixed failures was observed with increased etching periods, irrespective of the surface treatment after etching.*

Table 2: *Bond Strength Means–MPa (Standard Deviations) for All Groups*

Etching Period	Surface Treatment	
	Silane	Silane + Unfilled Resin
10 seconds	(G1) 19.4 (3.5)^{A,ab}	(G7) 17.4 (4.8)^{A,b}
20 seconds	(G2) 22.3 (5.1)^{A,a}	(G8) 21.3 (2.1)^{A,b}
40 seconds	(G3) 22.2 (3.2)^{A,a}	(G9) 21.1 (2.3)^{A,b}
60 seconds	(G4) 17.8 (2.1)^{B,b}	(G10) 24.7 (5.8)^{A,a}
120 seconds	(G5) 15.3 (3.0)^{B,bc}	(G11) 20.4 (2.2)^{A,b}
60+60 seconds	(G6) 14.3 (1.8)^{B,c}	(G12) 18.5 (4.6)^{A,b}

Means followed by distinct capital letters in the same line, and small letters in the same column, are significantly different at p<.05.

SEM Evaluation

In order to observe the topography of the conditioned surfaces, etched specimens (n=3) for each etching time were coated with gold and examined by scanning electron microscopy (SEM). Additionally, in order to observe the morphology at the bonding interfaces, ceramic-cement-ceramic sandwiched specimens were obtained for each group; two etched/silanated ceramic discs were bonded to each other using resin cement, similar to what has been previously described.[18] The specimens were embedded cross-sectionally in epoxy resin in order for the ceramic-cement interfaces to be viewed. After 24 hours, the specimens were wet-polished with 600-, 1200- and 2000-grit SiC paper followed by polishing with 3 µm, 1 µm and 0.5 µm diamond compounds. The cross-section profiles were examined by SEM, focusing on the depth of etching, micromechanical entanglement and integrity, homogeneity and continuity along the bonding interface.

RESULTS

Bond Strength and Failure Analysis

Results for the bond strength are shown in Table 2. The factors of the surface treatment and etching period were both significant, as was their interaction. Overall, the 60+60- and 120-second groups showed lower bond strengths than did the other groups. Samples treated with silane + resin (G7-G12) showed overall bond strengths higher than those of the groups only treated with silane. When only silane was applied, the groups etched for 20 and 40 seconds (G2 and G3) showed higher bond strengths than did the other groups, except for G1. In addition, the 60-second samples (G4) showed higher bond strengths than did the 60+60-second samples (G6). When silane + resin was applied, all etching periods showed similar results, except for the 60-second samples (G10), which presented higher bond strength.

Regarding a comparison between the surface treatments after etching, significantly higher bond strengths were observed for the application of silane + unfilled resin (G10-12) compared to silane only (G4-G6). The distribution of failure modes is shown in Figure 2. A decrease in adhesive failures and an increase in mixed failures was observed with the increased etching period. When comparing the different surface treatments, a decrease in adhesive failures was detected for the 10- and 20-second groups treated with silane + unfilled resin (G7 and G8) compared with the group treated with silane only (G1 and G2).

SEM Evaluation

SEM images of etched surfaces are shown in Figure 3. For the 10-second ceramic, poor dissolution of the vitreous phase was observed, with little exposure of the leucite crystals. For the 20-second ceramic, greater dissolution of the vitreous phase surrounding the leucite crystals was observed, although remnants of the unsolved vitreous phase were still present. For the ceramic etched for 40 seconds, the extent of dissolution was greater than for the ceramic etched for 20 seconds, causing extrusion of the crystals by detachment.

For the ceramic etched for 60 seconds, which is shown in Figure 3D, in addition to increased dissolution of the vitreous phase, fissures forming deep-grooved channels were observed. These fissures have limited extension and depth, and do not intercommunicate between themselves. For the 120-second sample (Figure 3E), intercommunication of the fissures and increased extent and depth of the channels were observed. The pattern of the deep-grooved channel formation is observed at even higher frequency and magnitude for the 60 + 60-second ceramic, and the intercommunication of fissures is extensive. SEM pictures of the cross-section profiles are shown in Figure 4. The samples etched by periods under 60 seconds showed full completion of the irregularities, even when only silane was applied. For the ceramic etched for 60 seconds and treated only with silane, the cement was generally able to penetrate the irregularities, although unfilled voids were sometimes detected. For the 120-second ceramic treated with only silane, discontinuation along the bonding interface was more frequent and extensive.

On the other hand, the unfilled voids on the ceramic etched for 60 seconds were completely infiltrated when the unfilled resin was applied. Likewise, the unfilled resin was able to infiltrate the irregularities on the 120-second ceramic (arrow on Figure 4E), contrasting with the profile when only silane was applied (pointer on Figure 4E); the higher magnification in Figure 4E shows the full infiltration of the unfilled resin on the 120-second ceramic. In contrast, even the unfilled resin was unable to completely penetrate the

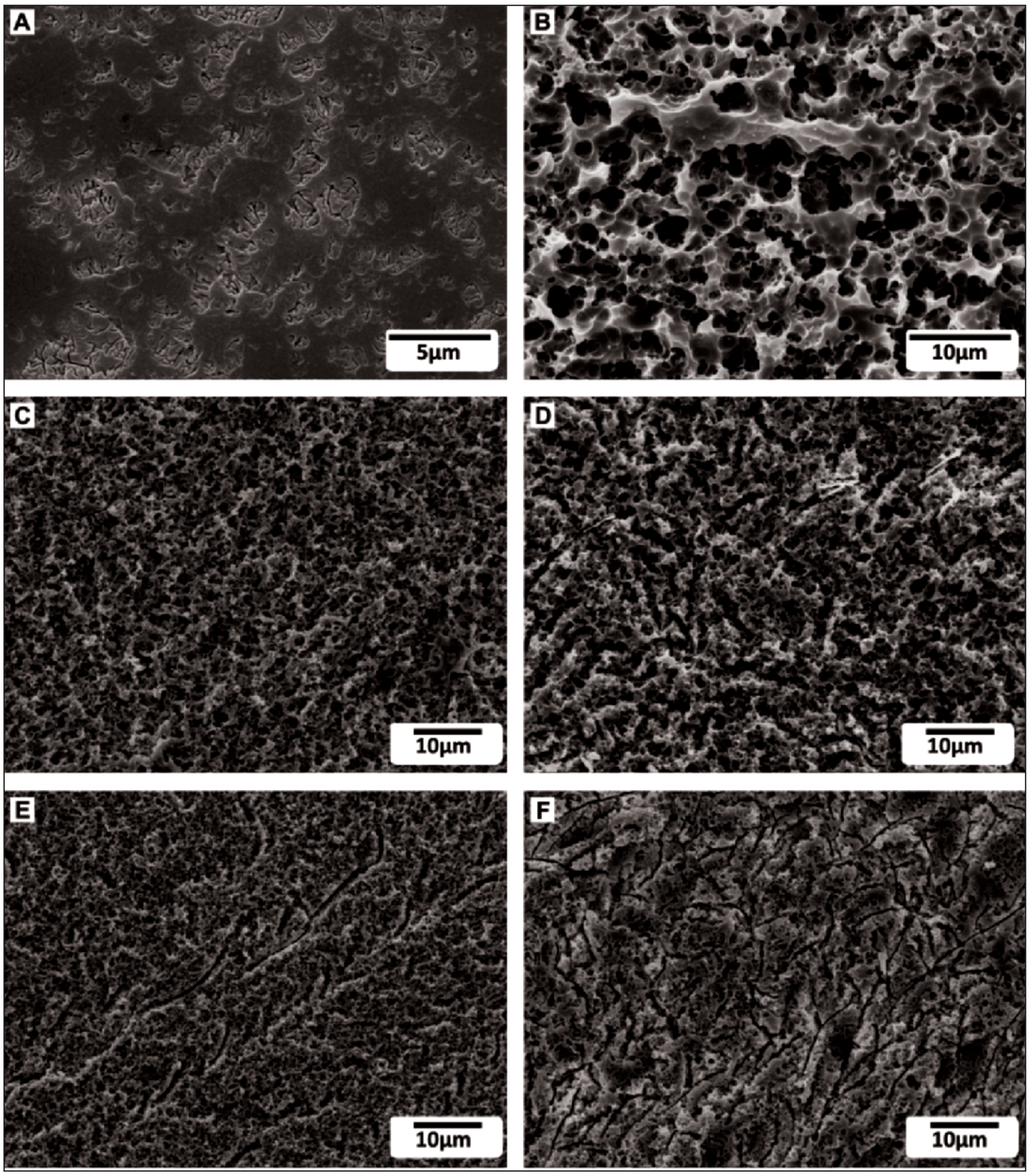

Figure 3: *SEM images of acid-etched surfaces. For the 10-second period (A), poor dissolution of the vitreous phase was observed, with little exposure of the leucite crystals. High dissolution of the vitreous phase, causing extrusion of the crystals by detachment, was observed for the 20-second (B) and 40-second periods (C), although dissolution was greater for 40 seconds. For the 60-second group (D), fissures forming deep-grooved channels were observed; these fissures have limited extension and depth and do not intercommunicate. For the 120-second sample (E), intercommunication of fissures and increased extent and depth of the channels was observed. The pattern of deep-grooved formation is observed in even higher frequency and magnitude for the re-etched ceramic (F) and the intercommunication of fissures is extensive.*

irregularities on the 60 + 60-second etched ceramic (Figure 4F).

DISCUSSION

The current results provide evidence that etching times higher than 60 seconds generally decreased the bonding ability to leucite-reinforced glass ceramic. Therefore, the tested hypothesis must be rejected. This result is in line with the findings of Barghi and others,[10] who reported decreased bond strengths for a medium-leucite-content ceramic when the samples were etched with HF gel for 90–180 seconds compared with 60 seconds. The increase in bond strengths with increased etching time might indeed occur, but only up to a certain level, after which increased etching times may decrease bond strengths. This might be explained by uncontrolled crystalline residue being deposited on the ceramic surfaces,[19-20] and the higher depth of dissolution promoted by the increased etching periods associated with poor penetration of the resin cement due to its high viscosity.

The cross-section profiles of the groups etched for 120 seconds and 60 + 60 seconds (G5 and G6) confirm insufficient penetration into the irregularities, leaving voids subjacent to the ceramic-cement entanglement (Figures 4E and 4F). Since ceramics are brittle,[11,21] the presence of unfilled areas may decrease their mechanical strength[22] by two main mechanisms: 1) the sharp geometry of the unfilled channels may create spots for stress concentration and 2) the fragile void area underneath the cement-ceramic entanglement may concentrate stress when submitted to mechanical loading. For the 120-second groups (G5 and G11), the grooved channels were observed less frequently and were less in magnitude than the re-etched samples (G6 and G12), suggesting limited interaction of the etchant, probably due to a combination of buffering and dilution of the HF by the crystalline residue during the longer exposure. When the ceramic is re-

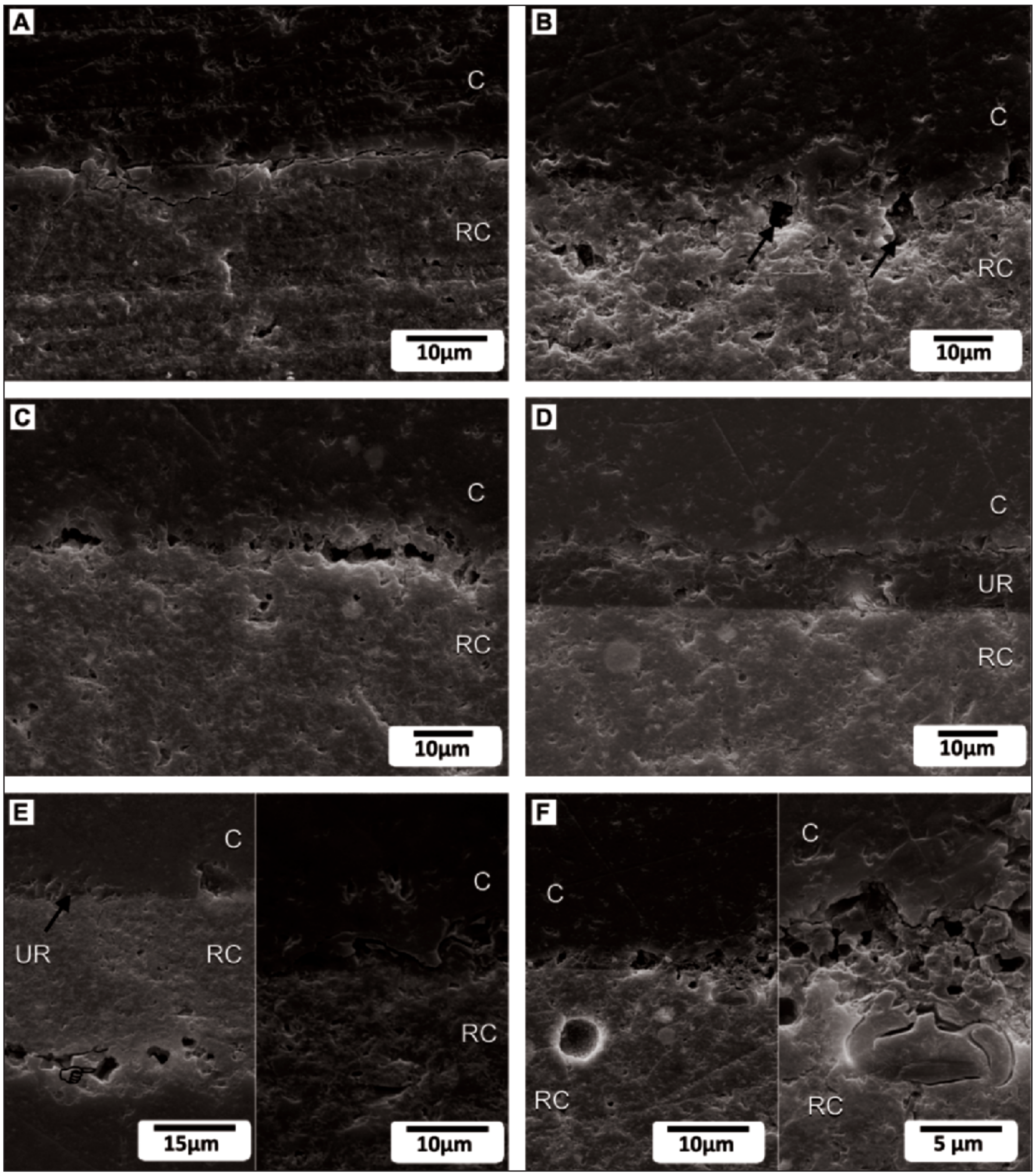

Figure 4: *SEM pictures of the cross-section profiles (C: ceramic; RC: resin cement; UR: unfilled resin). The 10-second sample (A) shows full completion of the irregularities. For G4, the cement was generally able to penetrate the irregularities, although unfilled voids were sometimes detected (arrows in B). For G5 (C), unfilled voids were more frequent and extensive. These unfilled voids were completely infiltrated on G10 (D). Likewise, the unfilled resin was able to infiltrate irregularities in the 120-second group (arrow on E), in contrast to the profile where only silane was applied (pointer on E); the higher magnification in Figure E shows full infiltration of the unfilled resin in the 120-second sample. In contrast, even the unfilled resin was unable to completely penetrate the re-etched ceramic (F).*

etched, the new HF exposure increases the etching aggressiveness and depth of dissolution. Although re-etching the ceramic is not indicated for cementation, this procedure might occur in clinical situations, for example, when the clinician reapplies the conditioning agent for increased retention.

When the unfilled resin was used, the bond strengths tended to be similar among the etching periods. In addition, when the unfilled resin was applied, higher bond strengths were observed for the 60-second, 120-second and 60 + 60-second groups (G10, G11 and G12) compared to specimens that were only silanated (G4, G5 and G6). Organosilane coupling agents are bifunctional molecules, with one end of the molecule capable of reacting with inorganic ceramic and the other end with the organic resin.[23] Nevertheless, silane merely forms a thin layer on the ceramic surface,[13] thus being unable to strengthen the etched structure by itself. The effectiveness of the bonding procedure using only silane depends on the ability to fill the irregularities with cement and close contact of the cement with the ceramic. On the other hand, the unfilled resin can both infiltrate the etched surface and entangle the substrate, reinforcing its structure.[24-25]

An increase in mixed failures was observed with prolonged conditioning periods when only silane was used; the depth of the dissolution is higher, and penetration of the cement, although not complete, is consequently higher. Therefore, there is an enhanced interlocking between these two substrates, generating failures that involve both. When both silane and unfilled resin were applied, the failures tended to be similar among groups, probably because of the better wetting of the resin onto the surface, thus infiltrating the irregularities created even by prolonged etching periods. Cross-section micrographs showed complete penetration of the irregularities by the unfilled resin, even for the 120-second group, although the re-etched ceramic still presented non-infiltrated voids.

The results of the current study have clinical implications. The 120- and 60 + 60- second etching periods promoted in-depth dissolution that the resin cement itself was unable to completely infiltrate. The application of a low-viscosity unfilled resin after silane might overcome poor infiltration, thus enhancing the bond strength even for re-etched ceramic. The internal surfaces of indirect restorations are not as well-polished as the specimens used in the current study, and this might create an even worse scenario for penetration. Therefore, application of an unfilled resin after silane should be encouraged in clinical practice. Furthermore, other variables should be evaluated in order to define the application of unfilled resin as a protocol for luting procedures, such as different cementation loads and viscosities of the luting material. Moreover, a non-solvated bonding resin was used, and it is unknown whether solvated agents would interfere with the silane layer. Finally, photo-activation of the unfilled resin before cementation should not be encouraged, as this procedure would increase the thickness of the bonding layer and interfere with adaptation of the ceramic restoration. Further studies focusing on these variables are necessary.

CONCLUSIONS

Increased etching periods generally decreased the bond strength to ceramic, while samples treated with both silane and unfilled resin showed bond strengths generally higher than the groups treated only with silane. Application of the unfilled resin over silane also provided better infiltration to the irregularities created on etched surfaces, irrespective of the conditioning time.

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