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Fatigue Failure Load of a Bonded Simplified Monolithic Feldspathic Ceramic: Influence of Hydrofluoric Acid Etching and Thermocycling

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Clinical Relevance

Defects introduced by hydrofluoric acid etching can propagate when the assembly is subjected to aging and fatigue stimuli, impairing its mechanical performance.

SUMMARY

Objective: To evaluate the effect of hydrofluoric acid (HF) etching and thermocycling (Tc) on fatigue failure load of feldspathic ceramic restorations cemented with two resin cements.

Methods: Disc-shaped feldspathic ceramic (Vitablocs Mark II; Ø=10 mm, 1.0-mm thick) and G10 epoxy resin (Ø=10 mm, 2.5-mm thick) specimens were made and randomly allocated considering three factors: ceramic etching (ie, with vs without 10% HF plus silane application), resin cement (ie, self-adhesive [RelyX U200; U200] or conventional [Multilink Automix; MA]), and Tc (ie, with vs without 5-55°C/12,000 cycles). Adhesive cementation followed each manufacturer's instructions. The fatigue test (n=20) was based on the staircase approach (250,000 cycles; 20 Hz). Contact angle, surface topography, and fractography analysis were also executed. Specific statistical tests were employed for each outcome (α=0.05).

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Results: The interaction of HF and Tc factors decreased the fatigue resistance for both cements (U200 542.63>U200/HF-Tc 495.00; MA 544.47>MA/HF-Tc 506.84). Comparing the cements associated with HF or Tc, there was statistical superiority for MA (U200-Tc 537.37<MA-Tc 561.32; U200/HF 535.79<MA/HF 557.11), and no statistical difference was detected when only cement type or its association with HF-Tc was compared (U200 542.63=MA 544.47; U200/HF-Tc 495.00=MA/HF-Tc 506.84). The fracture always originated from defects at the ceramic-intaglio surface as radial cracks.

Conclusion: HF etching plus silane agent increased the ceramic surface free energy and its wettability, but it did not provide better results in terms of fatigue resistance compared with silane agent application only. The association of HF etching and aging significantly reduced the fatigue resistance of the material, regardless of the resin cement used.

INTRODUCTION

Hydrofluoric acid (HF) etching followed by silanization is the most widely accepted surface treatment for glass ceramics.[1] HF etching selectively dissolves the glassy matrix of the glass-ceramic surface, creating retentive micropores, pits, cracks, and grooves on the conditioned surface, favoring micromechanical retention.[2,3] A silane coupling agent is applied on the etched surface to promote a chemical bond between the inorganic phase of the ceramic (ie, silica) with the organic phase of the resin cement (ie, polymer).[4–6]

However, this has become increasingly debatable, as some studies have also shown no need for HF etching for enhancing adhesion between resin cements and glass ceramics,[7–9] and it has also been demonstrated that HF etching may even lead to a ceramic weakening effect.[10,11] This deleterious impact is explained by the fact that HF etching also creates and increases the flaw population at the ceramic bonding interface. By that, it might increase the ceramic susceptibillity to slow crack growth of critical defects under the constant masticatory stresses, since the ceramic fracture strength is inversely proportional to the largest or critical flaw present in the loaded restoration, as described by Griffith's law.[12] From the clinical standpoint, the above-mentioned weakening effect is particularly relevant when considering the evidence showing that the glass-ceramic fails from flaws on the ceramic intaglio surface.[13,14]

Thus, a relevant factor to be considered as a predictor of the restoration performance seems to be the capacity of the cement to completely fill in the introduced defects,[15] which has been scarcely discussed in the literature. Resin cements are indicated for adhesive luting of feldspathic ceramic restorations. They provide better adhesion (higher bond strength) to both ceramic and restorative foundations/substrates as well as superior mechanical and optical properties (greater color selection possibilities and higher color stability), high resistance to hydrolysis, and great inherent tensile strength.[16] The advent of self-adhesive resin cements in the attempt to provide simplification and easy handling techniques appeared as a promising alternative approach, and these cements have shown acceptable esthetic properties and bond strength results that are at least comparable with the conventional resin cements.[1]

In addition to understanding the population of defects and the fill-up potential of different cements on feldspathic ceramic restorations, another important condition involved in such a scenario is the subsequent degradation of the bonding interface. According to Lu and others,[17] aging the bonding interface in water promotes a reduced elastic modulus of the cements as well as an apparent reduction in the bond strength. This degradation of cement properties may be sufficient to redistribute stresses in the restorative set, thus reducing the ability of restorations to tolerate masticatory loads over time.

Few studies have evaluated the mechanical performance of feldspathic ceramics cemented to tooth substrates or analog materials under fatigue stimuli.[18,19] According to Strasser and others,[20] ceramic pretreatments (eg, HF etching and air abrasion) are fundamental to provide a better and more stable adhesion (ie, reduced susceptibility to interface degradation) and consequently better performance and long-term predictability for such restorations, being that its absence may increase the risk of premature failure.

Thus, considering the above assumptions, the lack of consensus on this issue, and the clinical appeal, this laboratory study investigated the following: does HF etching change the flaw population on the glass-ceramic intaglio surface and consequently affect the fatigue behavior of adhesively bonded simplified monolithic glass-ceramic restorations when subjected to aging?

The study aimed to evaluate the influence of HF etching and aging on the fatigue failure load of a computer-aided design/computer-aided manufacturing (CAD/CAM) monolithic feldspathic ceramic adhesively bonded to a dentin analog material using two resin cements (a self-adhesive and a conventional). The assumed hypotheses were the following: 1) HF etching will not promote different fatigue behavior compared with nonetching, 2) thermocycling (Tc) will reduce the fatigue failure load results, and 3) no significant difference will be found between the cements.

METHODS AND MATERIALS

Study Design

Restorative sets were fabricated through a simplified approach[3] using feldspathic ceramic discs (Vita Mark II) cemented to glass fiber reinforced epoxy resin discs (G10 substrate, dentin analog) with conventional (MA; Multilink Automix, Ivoclar Vivadent, Schaan, Liechtenstein) and self-adhesive (U200; RelyX U200, 3M ESPE, Seefeld, Germany) resin cements. The diameter of the specimens was determined as 10.0 mm because it resembles the average diameter of the first molar occlusal surface,[21] and the final thickness of the cemented set was 3.5 mm because it is equivalent to the average thickness between the occlusal surface and the pulp chamber roof of molar teeth.[22,23] The specimens were prepared and randomly (www.random.org) distributed considering the three factors under study: ceramic etching (ie, with vs without 10% HF etching plus silane application), resin cement (ie, self-adhesive [U200] or conventional [MA]), and Tc (ie, with vs without; 5-55°C/12,000 cycles; Table 1).

Table 1: *Study Experimental Design*

Resin Cement (Manufacturer)	Baseline		Aged[a]	
	No HF[c]	HF[b,c]	No HF[c]	HF[b,c]
RelyX U200 (3M ESPE)	U200	U200/HF	U200-Tc	U200/HF-Tc
Multilink Automix (Ivoclar Vivadent)	MA	MA/HF	MA-Tc	MA/HF-Tc

[a] *Thermocycling (Tc): 12,000 cycles between 5 and 55°C.*
[b] *Ten percent hydrofluoric acid etching for one minute.*
[c] *Silane coupling agent application: RelyX Ceramic Primer for the RelyX U200 cement and Monobond Plus for the Multilink Automix cement.*

Production of Ceramic Discs

Prefabricated feldspathic ceramic blocks (Vitablocs Mark II for CEREC/inLab, Lot 45950, Vita Zahnfabrik, Bad Säckingen, Germany) were rounded into cylinders with a diamond drill (internal Ø=10 mm; Diamant Boart, Brussels, Belgium) coupled to an electric drill (SBE 1010 Plus, Metabo, Nürtingen, Germany) and then sectioned in a precision diamond saw (Isomet 1000, 15LC Diamond Disc, Buehler, Lake Bluff, IL, USA), resulting in disc-shaped specimens of 1.05 mm thickness. The procedures were executed with constant and abundant water cooling. The occlusal surfaces of the discs were sequentially polished (#400-, #600-, #800-, #1200-, and #2000-grit silicon carbide abrasives; Buehler) to obtain a smooth top surface, removing the defects introduced by cutting and obtaining a final thickness of 1.0 mm (±0.01 mm). The opposite surface (ie, cementation/intaglio surface) was kept as cut to resemble milled CAD/CAM surfaces. After, the discs were cleaned with isopropyl alcohol (78%; 10 minutes) in an ultrasonic bath (1440 D, 50/60 Hz, Odontobras, Ribeirão Preto, Brazil) to remove any processing sediment (cutting and polishing procedures).

Production of Dentin Analog Discs

Epoxy resin cylinders (±250-mm length×12.7-mm Ø; NEMA grade G10, Accurate Plastics Inc, New York, NY, USA) were rounded in a polishing machine (200- and 600-grit silicon carbide abrasives; EcoMet/AutoMet Polisher, Buehler) to obtain cylinders of 10 mm in diameter and then cut with a precision diamond saw (Isomet 1000, Buehler), as previously described in the section "Production of Ceramic Discs," with the exception that the thickness of this material was set to 2.7 mm. Both sides of the discs were sequentially polished (#400- and #600-grit SiC abrasives) to obtain smooth surfaces, removing the defects introduced by cutting until a final thickness of 2.5 mm was achieved.

Cementation Procedure

Prior to cementation procedures, the discs were randomly assigned into eight groups (n=25) according to the study factors (Table 1). Based on the two levels (with vs without) considered in the HF etching factor, half of the ceramic specimens were submitted only to an ultrasonic bath (distilled water, five minutes) and air dried for 30 seconds, remaining untouched until the cementation procedure, while the other half was etched with 10% HF (Condac 10 Porcelana, FGM, Joinville, Brazil) for one minute, rinsed (one minute), submitted to the ultrasonic bath protocol to remove any precipitates generated from acid etching, and then air dried for 30 seconds prior to the cementation procedure. The epoxy resin specimens were all etched with 10% HF (Condac 10

Porcelana, FGM) for one minute, rinsed (one minute), submitted to an ultrasonic bath (distilled water, five minutes) to remove the debris formed during acid etching, and air dried for 30 seconds prior to the cementation procedure.

The other cementation procedures were performed according to the manufacturer's instructions of the respective cement:

- Self-adhesive resin cement (U200): The silane-based primer (RelyX Ceramic Primer, 3M ESPE) was scrubbed in both substrates (ceramic and epoxy resin) for five seconds and air-dried until the solvent evaporation (10 seconds).
- Conventional resin cement (MA): Ceramic conditioning: The silane-based primer (Monobond Plus, Ivoclar Vivadent) was applied on the ceramic bonding surface, scrubbed for 15 seconds, then left to react for 45 seconds, and air dried. Epoxy resin conditioning: After the aforementioned HF etching, Multilink Primers A and B (Ivoclar Vivadent) were mixed in a 1:1 ratio, scrubbed on the epoxy resin surface for 30 seconds, and air dried for about five seconds to obtain a thin film.

After the primer applications, the respective cement was mixed (1:1 ratio) and applied on the epoxy resin, and the ceramic disc was seated under a load of 250 g. The cement excess was removed with a Microbrush, and light-curing was performed (1200 mW/cm^2; Radii-cal, SDI Limited, Bayswater, Australia) for 20 seconds at each region (occlusal and four axial surfaces: 0, 90, 180, and 270°). Prior to the fatigue test, the specimens were stored in distilled water (37°C) for four days. The specimens that were thermocycled were stored (distilled water, 37°C) for one day prior to the Tc and for an additional four days after Tc and prior to the fatigue test.

Thermocycling

The Tc subgroups (Table 1) were subjected to aging by Tc to stimulate the degradation of the bond interface. The specimens were subjected to 12,000 thermal cycles in water at temperatures of 5°C and 55°C, with a dwell time of 30 seconds and a transfer time of four seconds (Ethik Technology, Vargem Grande Paulista, São Paulo, Brazil).

Fatigue Failure Load Test: Staircase Method

First, to define the fatigue test parameters for each condition, a load-to-fracture test was executed in a universal testing machine (n=5; EMIC DL 2000, São José dos Pinhais, SP, Brazil) with a crosshead speed of 1 mm/min and incremental load until the auditory perception of cracking (ie, presence of radial cracks confirmed by light trans-illumination and visual inspection) by a single trained blinded operator (L.F.G.; ie, the researcher did not know the respective group at the moment of testing and inspection).

The fatigue test was run in an electric machine (Instron ElectroPuls E3000, Instron Corp, Norwood, MA, USA) following the staircase method described by Collins.[24] To better distribute the stress during testing and to avoid contact damage (Hertzian's cone cracks; fracture by surface contact damage), an adhesive tape (110 μm) was placed on the feldspathic top surface, and a nonrigid sheet (cellophane; 2.5 μm) was placed between the piston and the specimen.[19,25,26] The specimens were placed on a flat steel base and submerged in distilled water, and a stainless-steel hemisphere of 40 mm in diameter was used to apply the load on the center of the specimens' top surface.[14,27,28]

The fatigue test (n=20; staircase method) was run under a frequency of 20 Hz during 250,000 load pulses in each step, with a load amplitude ranging from a minimum of 10 N to the maximum load to failure for each specimen. The first specimen from each group was tested with an initial load close to the estimated fatigue failure load (~60% of the mean of load-to-fracture test) until the fracture or survival was observed at the number of predetermined cycles (250,000). Then, the next specimen was tested with a step size (~5% of initial load) higher (when the previous specimen survived) or lower (when the previous specimen failed) than the initial loading level. This procedure was repeated until at least 15 samples per group were tested after the start of the test, and according to Collins,[24] the test starts only after the first stair inversion (first different outcome obtained), with 15 specimens being required to get reliable results following this methodology.

Contact Angle Measurements

Additional ceramic samples (n=3) were obtained, treated for each evaluated condition (HF etching plus silane coupling agent application; HF application only; silane application only; and baseline, no HF and no silane application), and subjected to contact angle analysis through the sessile drop technique using a goniometer (Drop Shape Analysis, model DSA 30S, Krüss, Hamburg, Germany) connected to a software program (DSA3, V1.0.3-08, Krüss). A drop (11 μL) of deionized water was deposited on the ceramic-treated surface using a

Table 2: *Results From the Monotonic Load-to-Failure Test (n=5)*[a]

Group	Mean of Load-to-Failure Test (N)	Initial Load for Fatigue Test (N)	Step Size (N)	Mean Load for Fatigue Failure, L_f (SD) (N)[b]
U200	820.4	490	25	542.63 (21.88) Ab
U200-Tc	900.8	540		537.37 (21.88) Ab
U200/HF	863.9	520		535.79 (22.38) Ab
U200/HF-Tc	870.6	520		495.00 (22.05) Bb
MA	841.0	505	25	544.47 (28.03) Ab
MA-Tc	888.5	535		561.32 (41.23) Aa
MA/HF	897.8	540		557.11 (20.50) Aa
MA/HF-Tc	781.1	470		506.84 (42.79) Bb

[a] *The parameters defined to start the fatigue test (n=20; initial load for fatigue test=~60% from the mean of load to fracture; step size=~5% of initial load for fatigue test) based on the staircase method and mean load for fatigue failure (L_f and standard deviation [SD]) obtained through the fatigue test.*
[b] *Different uppercase letters indicate statistical differences depicted by one-way ANOVA and post hoc Bonferroni tests considering each cement individually (α=0.05). Different lowercase letters indicate statistical differences depicted by independent-samples* t*-tests for paired conditions between both cements (U200 vs MA,* p*=0.823; U200-Tc vs MA-Tc,* p*=0.034; U200/HF vs MA/HF,* p*=0.004; U200/HF-Tc vs MA/HF-Tc,* p*=0.293; α=0.05).*

syringe, and five seconds after dropping, the contact angle was measured for 10 seconds (series of 30 images per second).

Topographic Analysis

Additional ceramic samples (n=2) for each evaluated condition (with and without HF etching) were prepared as previously described, sputtered with a gold-palladium alloy under vacuum, and then examined under scanning electron microscopy (SEM; VEGA3 Tescan, Brno-Kohoutovice, Czech Republic) to evaluate their surface topography (500× and 2500× magnification) and defects created by HF etching in the cross-sectional view (500× and 3500× magnification). SEM images were obtained through the use of two detectors: secondary electron and back-scattering electron.

Fractographic Analysis

All failed specimens after the fatigue test were evaluated in a stereomicroscope (Stereo Discovery V20, Carl-Zeiss, Gottingen, Germany) to determine the presence and direction of radial cracks by light transillumination. Next, these specimens were sectioned in two halves, perpendicular to the direction of the cracks, in a high-precision diamond saw (Isomet 1000, Buehler). Then, the sectioned halves were reanalyzed in a stereomicroscope to determine the crack origin and its propagation direction, and the representative cracks were selected and sputtered with a gold-palladium alloy under vacuum for a descriptive analysis of higher resolution in SEM (as described for topographic analysis) with 1000× and 2500× magnification.

Data Analysis

All statistical analyses were performed using the IBM SPSS Statistics Program (v24 for Windows; IBM Corp; α=0.05).

First, a three-way analysis of variance (ANOVA) was used to determine the influence of each factor on the fatigue failure load of the restorative set and to elucidate any presence of interaction between the independent study variables (cement, HF etching, and Tc).

One-way ANOVA and post hoc Bonferroni tests were adopted to compare the effect of different conditions for each respective cement separately. *t*-Tests for independent samples were used between paired conditions (U200 vs MA; U200-Tc vs MA-Tc; U200/HF vs MA/HF; U200/HF-Tc vs MA/HF-Tc) to compare and depict the statistical differences for fatigue failure load between the cements exposed to the same study factor.

RESULTS

Fatigue Failure Load Test: Staircase Method

Based on the three-way ANOVA, there were statistically significant influences of the factors cement (p=0.006; MA>U200), Tc (p=0.000; without Tc>with Tc), and HF etching (p=0.000; without HF>with HF) and for the interaction HF × Tc (p=0.000) on the fatigue load results. No statistically significant influence was detected for other associations.

Analyzing each cement separately, the one-way ANOVA and post hoc Bonferroni analyses showed that both cements behaved similarly to the different

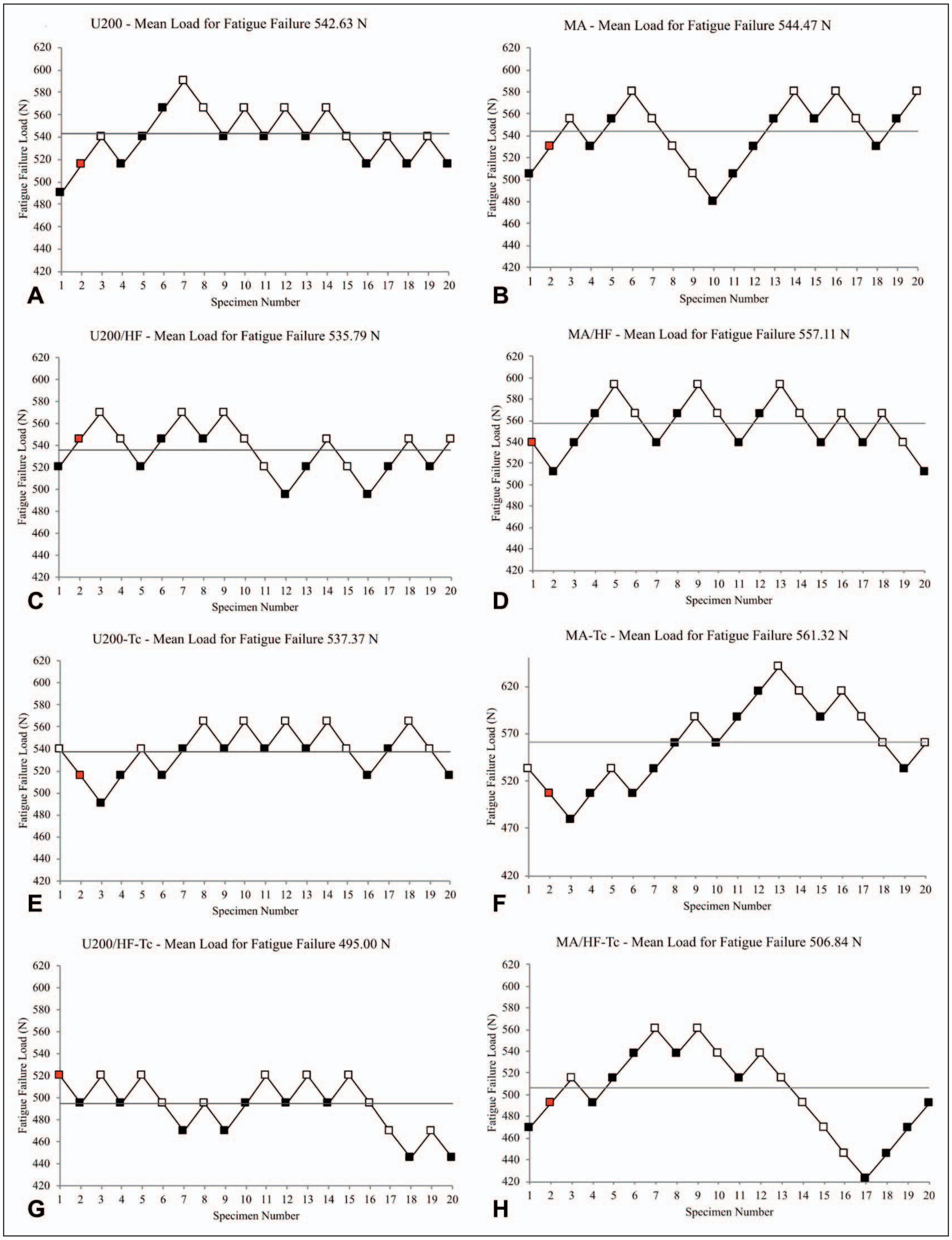


Figure 1. *Survival and failure patterns observed during staircase fatigue testing (250,000 cycles; 20 Hz). Horizontal lines indicate the mean load value, red marks the start of up-and-down characters, and solid marks represent survival and empty marks represent failure.*

conditions, being that the fatigue resistance was statistically reduced only when the HF and Tc factors were applied together (Table 2; Figure 1).

Comparing the same conditions between the cements through *t*-tests for independent samples, the MA cement yielded better results when the specimens were submitted to HF etching or Tc factors. There was no difference between the cements at baseline (without HF and without Tc) or for the association of factors (with HF and Tc; Table 2).

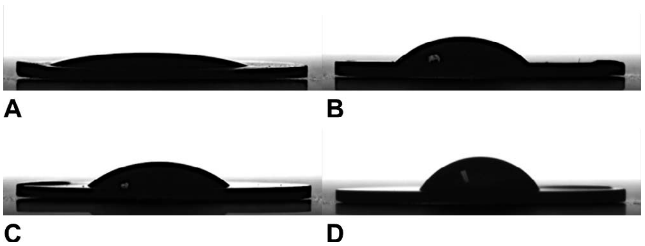


Figure 2. *Representative images and results of contact angle measurements of ceramic surfaces subjected to the following treatments: (A): Hydrofluoric acid etching (HF) plus silane coupling agent application (10.45±2.94). (B): Only HF application (44.15±13.70). (C): Only silane application (33.27±3.55). (D): Baseline (no treatment) (56.24±1.71).*

Contact Angle Measurements

The lowest contact angle was observed after HF etching plus silane application, indicating greater surface wettability. The highest contact angle (lowest wettability) was observed on the untreated surface (baseline group). Intermediate values were observed when HF etching only or silane only was used (Figure 2).

Topographic Analysis

Topographic and cross-sectioned SEM images of baseline (nonetched) and HF-etched (HF 10%; one minute) specimens are shown in Figure 3. Etched surfaces presented an irregular topography characterized by the presence of numerous micro irregularities, pits, grooves, and striations as a result of the glassy phase dissolution, known as a honeycomb-etched pattern (Figure 3A). The baseline images showed a smoother and more homogeneous surface without any relevant irregularity (Figure 3B).

Fractographic Analysis

Stereomicroscope and SEM analysis showed that all failures were radial cracks starting from the ceramic intaglio surface (Figure 4). Cracks due to contact damage between the piston and the ceramic surface were not found.

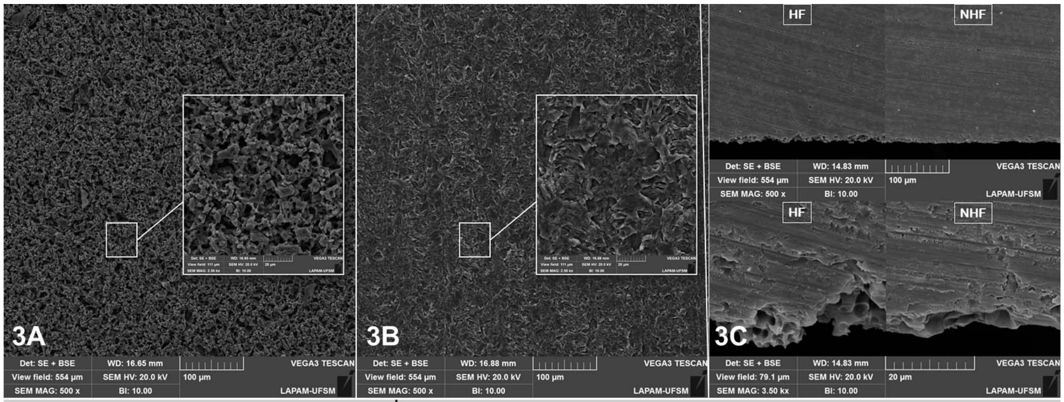
DISCUSSION

Our findings show that the HF etching condition associated with Tc had a significant deleterious effect on the load for fatigue failure regardless of the cement used, and HF etching prior to silanization did not enhance the fatigue resistance of the simplified ceramic restorations.

HF etching promotes a surface dissolution of the ceramic glass matrix, creating micro retentions that contribute to the mechanical bonding with dental substrate when adhesively bonded.[9,10,29] However, this has become debatable, since fatigue properties of all-ceramic systems can be related to the flaw population (size, number, and distribution) of the material,[12] and some studies have demonstrated that HF etching may lead to a decrease in the feldspathic ceramics strength by introducing defects on the surface that may not be completely filled by the resin cement.[10,30,31] In our study, the first hypothesis was accepted since HF etching did not promote different fatigue failure load results compared with baseline. Regarding the Tc factor, the second hypothesis was partially accepted since it reduced the fatigue failure load only for the HF-etched specimens.

Other authors have obtained similar results, corroborating that HF etching improves the bond strength by creating micromechanical interlocking but may lead to a weakening effect on the ceramic, thereby compromising the clinical performance of the restoration.[10,31] Moreover, recent studies corroborate these findings, showing worse or equal results for bond strength,[7] biaxial flexural strength,[11] and fatigue load[19] when conditioning glass-ceramic with HF prior to bonding, proving that this subject still requires future evaluations and considerations. In addition, HF has potentially hazardous toxicity known from other applications and these risks should also be considered when applied in dentistry.[32]

The topographic images in Figure 3C show the micro retentions, pits, and grooves created by the selective HF etching of the glass-ceramic vitreous matrix, which could lead to a decrease in the ceramic fatigue resistance, especially when the defects are not completely filled by the cement. Defects created by HF etching may also lead to stress concentration, which can result in ceramic premature fracture starting from the adhesive interface,[18,30] as observed in our study (Figure 4). This behavior is particularly important since sharp defects (as created by HF etching; Figure 3C) are more damaging than rounded defects. In this sense, it has to be emphasized that we used a high-concentration HF etchant (10% HF for one minute), which produces many more defects than in lower concentrations (for instance, 1% and 5%)[19] and makes the material more susceptible to the presence of unfilled defects after bonding and, consequently, to crack initiation and propagation under fatigue loads. Clinical[33,34] and laboratory studies[18] on failed glass-ceramic crowns have reported that the great majority of bulk fractures start from flaws on the ceramic-intaglio surface, where high tensile stress is concentrated.



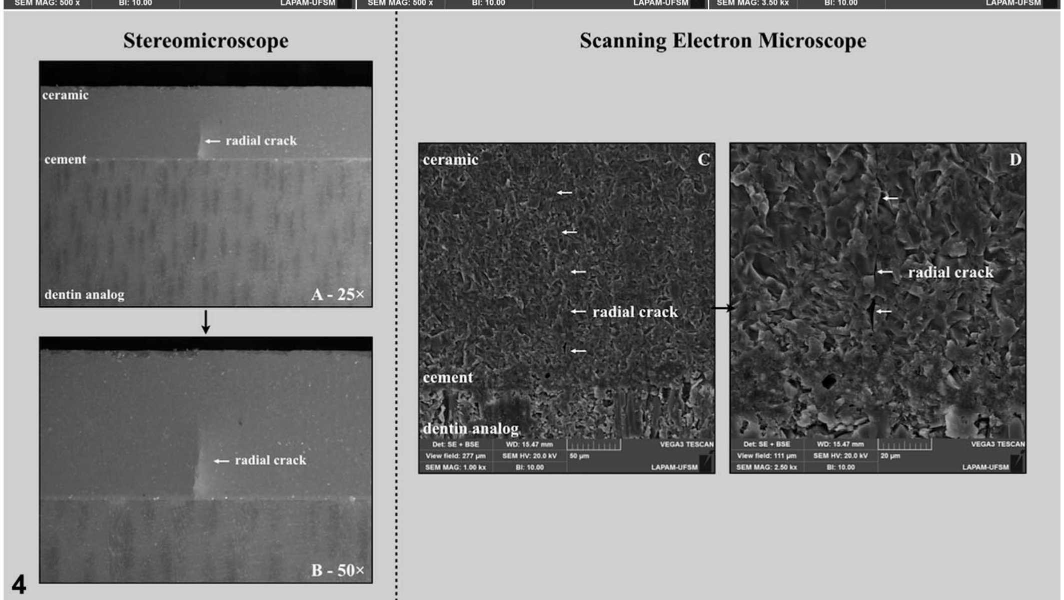


Figure 3. *SEM images of the ceramic surface with hydrofluoric acid (HF) etching (A: 500× and 2500×) and without hydrofluoric acid etching (B: 500× and 2500×). (C): Etching pattern in cross-sectioned specimens: on the left side is the HF-etched ceramic in 500× (top left) and 3500× (bottom left) magnification; on the right side is the nonetched (NHF) ceramic in 500× (top right) and 3500× (bottom right) magnification.*

Figure 4. *Radial crack was the failure pattern for the failed specimens in all groups: The images show the radial crack that indicates the origin and pattern of ceramic failure. (A, B): Stereomicroscope images of cut representative specimens where we can clearly see the radial crack (white arrows) starting at the intaglio-ceramic surface. (C): 1000× and (D): 2500× magnification: SEM images of a cut representative specimen with white arrows pointing to the radial crack.*

Adhesive cementation significantly increases the restorative material's fracture loads.[35] Using finite element analysis and fatigue testing for the lithium disilicate glass-ceramic, de Kok and others[36] proved that proper adhesion can better distribute stress during loading, increasing the material's resistance. In our study, the group treated with HF (which provides a surface with many more defects; Figure 3) had a statistically similar fatigue resistance to the nonetched surface (baseline; Table 2), corroborating the results found by de Kok and others[36] of the protective role of bonding in overcoming the ceramic's internal roughness effect.

Regarding the resin cement filling capacity, cements with low viscosity are more prone to penetrate the ceramic than cements with high viscosity.[37] According to Gamal and others,[38] self-adhesive resin cements have high viscosity and consequently are less capable of spreading onto the ceramic and substrate surfaces, which may compromise its wettability. The application of HF etching plus silane has been recommended to promote chemical bonding between inorganic molecules of the ceramic with organic molecules of these resin cements.[39,40] This sequence is already well established in the literature in terms of bond strength, as these procedures increase the surface energy of the ceramic and the wettability of the resin cement, improving adhesion.[3,31] This greater wettability can be evidenced by the lower contact angle of the treated surfaces, as shown in Figure 2A. In our work, in terms of fatigue resistance, we can note that this procedure may not summarily be necessary, since the groups that were not submitted to HF etching had the same or even better results than the etched ones. However, it must be considered that this assertion is a finding only with respect to fatigue resistance. From this viewpoint, there is a need to develop novel surface-conditioning methods to address the problem related to the bond durability and defect fill-in potential[6] and to try to eliminate the use of HF to produce a less technique-sensitive and safer (ie, using a less hazardous material) bonding system.

As the conventional cement performed better than the self-adhesive when the HF etching and Tc factors were applied separately, the third hypothesis was partially accepted. According to Gamal and others,[38] that result could be explained by the high viscosity of the self-adhesive resin cement and less spreadability onto the substrate-ceramic surface, reducing its ability to infiltrate into surface irregularities.

When the materials are free to deform, they will expand or contract due to fluctuations in temperatures. By that, the temperature change during Tc is a deleterious factor for the adhesion between ceramic and dental substrate.[41] Because of the different coefficients of linear thermal expansion between the materials in the adhesive interface (ceramic/cement/substrate), they have different degrees of contraction and expansion during Tc, leading to micromechanical fatigue stresses in the adhesive interface, breaking adhesive bonds, and finally reducing the adhesion quality.[41] In the present study, aging (Tc 5-55°C/12,000 times) significantly affected the fatigue resistance for both cements only when the ceramic was previously etched with HF. This corroborates the findings of Venturini and others,[31] who hypothesized that when the micro retentions are not completely filled by the cement, this empty and unfilled space allows faster water absorption at the interface of the restoration with its consequent hydrolysis and degradation and finally a decrease in the material fatigue resistance.

The present study implemented a fatigue test under a wet environment, where constant loads with ranging intensity were applied until the failure of the specimens. This method mimics the oral environment and more closely simulates the masticatory stresses when compared with the static test.[35] However, the applied test setup (axial load) may not fully simulate all the forces to which the material is subjected in the oral environment, especially to lateral loads (sliding motion) that generate compressive, tensile, and shear stresses on the ceramic surface leading to the subsurface crack formation and propagation.[42] Another limitation may be the simplified restorative set (disc-shaped specimens), which does not completely simulate the anatomy of a molar crown.

In our study, the loads at initial radial cracks (overall mean equal to 534.83 N) exceeded the maximum bite forces during mastication (148.73 to 354.01 N)[43] but were far below the maximum bite forces reached in sleep associated bruxism (±800 N) and maximum voluntary bite forces during daytime (±1000 N).[44] Considering the subjects discussed above, we emphasize that our results should be carefully analyzed, and more *in vitro* and clinical findings can corroborate our results.

CONCLUSIONS

- The HF etching associated with a silane coupling agent increased the ceramic surface free energy (lower contact angle and consequently higher wettability), but it did not provide better results in terms of fatigue resistance compared with silane agent application only.
- When the feldspathic ceramics were HF etched, bonded (regardless of the cement used), and then subjected to aging, the fatigue failure load of the restorations was significantly reduced.
- HF etching and Tc factors applied alone did not lead to degradation of the ceramic fatigue resistance, regardless of the resin cement used (conventional [MA] or self-adhesive [U200]), being that the conventional cement performed better in both cases (only HF and only Tc).

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Conflict of Interest

The authors have no financial interest in any of the companies or products mentioned in this article.

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